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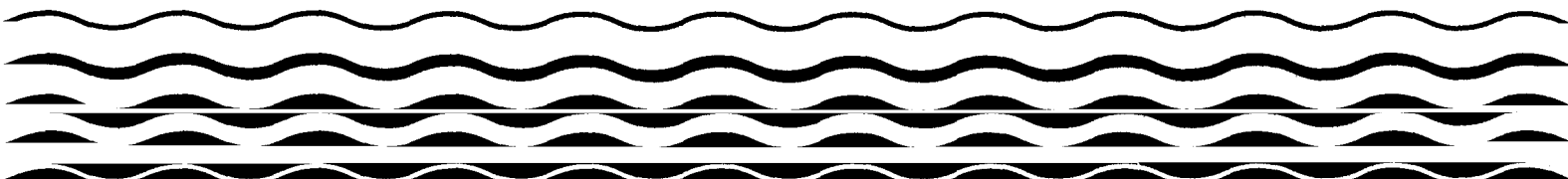
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# **Guide for Conducting Treatability Studies under CERCLA Solvent Extraction**

## **Interim Guidance**



**GUIDE FOR CONDUCTING  
TREATABILITY STUDIES UNDER CERCLA:  
SOLVENT EXTRACTION  
INTERIM GUIDANCE**

U.S. Environmental Agency  
Risk Reduction Engineering Laboratory  
Office of Research and Development  
Cincinnati, Ohio 45268

and

Office of Emergency and Remedial Response  
Office of Solid Waste and Emergency Response  
Washington, D.C. 20460



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## **DISCLAIMER**

The information in this document has been funded wholly or in part by the U.S. Environmental Protection Agency (EPA) under contract No. 68-C8-0062, Work Assignment 3-23, to Science Applications International Corporation (SAIC). It has been subjected to the Agency's peer and administrative reviews and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

# FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

The primary purpose of this guide is to provide standard guidance for designing and implementing a solvent extraction treatability study in support of remedy selection. Additionally, it describes a three-tiered approach, that consists of 1) remedy screening, 2) remedy selection, and 3) remedy design, to solvent extraction treatability testing. It also presents a guide for conducting treatability studies in a systematic and stepwise fashion for determination of the effectiveness of solvent extraction (in conjunction with other treatment technologies) in remediating a CERCLA site. The intended audience for this guide comprises Remedial Project Managers (RPMs), On-Scene Coordinators (OSCs), Potentially Responsible Parties (PRPs), consultants, contractors, and technology vendors.

E. Timothy Oppelt, Director

Risk Reduction Engineering Laboratory

# ABSTRACT

Systematically conducted, well-documented treatability studies are an important component of the remedial investigation/feasibility study (RI/FS) process and the remedial design/remedial action (RD/RA) process under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). These studies provide valuable site-specific data necessary to aid in the selection and implementation of the remedy. This manual focuses on solvent extraction treatability studies conducted in support of remedy selection prior to developing the Record of Decision.

This manual presents a standard guide for designing and implementing a solvent extraction remedy selection treatability study. The manual describes and discusses the applicability and limitations of solvent extraction technologies, and defines the prescreening and field measurement data needed to determine if treatability testing is required. It also presents an overview of the process of conducting treatability tests and the applicability of tiered treatability testing for evaluating solvent extraction technologies. The specific goals for each tier of testing are defined and performance levels are presented, which should be met at the remedy screening and remedy selection levels before additional tests are conducted at the next tier. The elements of a treatability study work plan are also defined with detailed discussions on the design and execution of the remedy screening and remedy selection treatability studies.

The manual is not intended to serve as a substitute for communication with experts or regulators nor as the sole basis for the selection of solvent extraction as a particular remediation technology. Solvent extraction must be used in conjunction with other treatment technologies since it generates residuals. This manual is designed to be used in conjunction with the Guide for Conducting Treatability Studies Under CERCLA (Interim Final).<sup>(27)</sup> The intended audience for this guide comprises Remedial Project Managers (RPMs), On-Scene Coordinators (OSCs), Potentially Responsible Parties (PRPs), consultants, contractors, and technology vendors.

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William McGovern	CF Systems
Steve Schwartz	Versar
Jean Paquin	Sanivan Group
Avijit Dasgupta	ABB Environmental
Saeed Darian	Nukem Development
Lanny Weimer	Resources Conservation Company
Barry Rugg	ART International
Monique Punt	Environmental Canada
C. Judson King	University of California, Berkeley
G. Bradley Hunter	Tennessee Valley Authority
John Napier	Martin Marietta
Rodney Hodgson	Hazen Research Group
Alan Cash	Terra-Kleen
Scott Engle	PRC Environmental
André Zownir	U.S. EPA, ERT
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# SECTION 1

## INTRODUCTION

### 1.1 BACKGROUND

Section 121(b) of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) mandates EPA to select remedies that “utilize permanent solutions and alternative treatment technologies or resource recovery technologies to the maximum extent practicable” and to prefer remedial actions in which treatment that “permanently and significantly reduces the volume, toxicity, or mobility of hazardous substances, pollutants, and contaminants is a principal element.” Treatability studies provide data to support treatment technology selection and remedy implementation. If treatability studies are used, they should be performed as soon as it is evident that insufficient information is available to ensure the quality of the decision. Conducting treatability studies early in the remedial investigation/feasibility study (RI/FS) process reduces uncertainties associated with selecting the remedy and provides a sound basis for the Record of Decision (ROD). EPA Regional planning should factor in the time and resources required for these studies.

Treatability studies conducted during the RI/FS phase indicate whether the technology can meet the cleanup goals for the site, whereas treatability studies conducted during the remedial design/remedial action (RD/RA) phase establish design and operating parameters for optimization of technology performance. Although the purpose and scope of these studies differ, they complement one another since information obtained in support of remedy selection may also be used to support the remedy design.<sup>(38)</sup>

This document refers to three levels or tiers of treatability studies: remedy screening, remedy selection, and remedy design. Three tiers of treatability studies are also defined in the Guide for Conducting Treatability Studies Under CERCLA, Interim Final,<sup>(27)</sup> hereinafter referred to as the “generic guide”. The generic guide refers to the three treatability study tiers, based largely on the scale of test equipment, as laboratory screening, bench-scale testing, and pilot-scale testing. Laboratory screening is typically used to screen potential remedial technologies and is equivalent to remedy screening. Bench-scale testing is typically used for remedy selection; however it may fall short of providing enough information for remedy selection. Bench-scale studies can, in some cases, provide enough information for full-scale design. Pilot-scale studies are normally used for remedial design, but may be required for remedy selection in some cases. Because of

the overlap between these tiers, and because of differences in the applicability of each tier to different technologies, the functional description of treatability study tiers (i.e., remedy screening, remedy selection, and remedy design) has been chosen for this document.

Some or all of the treatability study levels may be needed on a case-by-case basis. The time and cost necessary to perform the testing are balanced against the improved confidence in the selection of treatment alternatives. These decisions are based on the quantity and quality of data available and on other factors (e.g., state and community acceptance of the remedy, additional site data and experience with the technology). The need for each level of treatability testing required are management decisions. Section 3 discusses using treatability studies in remedy evaluation in greater detail.

### 1.2 PURPOSE AND SCOPE

This guide helps ensure a reliable and consistent approach in evaluating whether solvent extraction should be considered for site remediation. This guide discusses the remedy screening and remedy selection levels of treatability testing. Remedy screening studies provide a quick and relatively inexpensive indication of whether solvent extraction is a potentially viable remedial technology. The remedy selection treatability test provides data to help determine if reductions in contaminant volumes will allow cost-effective treatment to meet site cleanup goals. Remedy selection studies also provide a preliminary estimate of the cost and performance data necessary to design either a remedy design study or a full-scale solvent extraction system. While solvent extraction technology may be applicable to inorganic contaminants in some instances, the primary use of solvent extraction, and therefore the focus of this guide, concerns the treatment of organic contaminants.

In general, remedy design studies will also be required to optimize full-scale system design. Presumably, before remedy design studies are conducted, it has already been decided that solvent extraction is an economically and technically viable treatment alternative with remedy selection testing. Remedy design is not discussed in this guidance document.

### 1.3 INTENDED AUDIENCE

This document is intended for use by Remedial Project

Managers (RPMs), On-Scene Coordinators (OSCs), Potentially Responsible Parties (PRPs), consultants, contractors, and technology vendors. Each has different roles in conducting treatability studies under CERCLA. Specific responsibilities for each can be found in the generic guide.<sup>(2)</sup>

## 1.4 USE OF THIS GUIDE

This guide is organized into seven sections and reflects the basic information required to perform treatability studies during the RI/FS process. Section 1 is an introduction which provides background information on the role of the guide and outlines its intended audience. Section 2 describes different solvent extraction processes currently available and discusses how to conduct a preliminary screening to determine if solvent extraction is a potentially viable remediation technology. Section 3 provides an overview of the different levels of treatability testing and discusses how to determine the need for treatability studies. Section 4 provides an overview of the remedy screening and remedy selection treatability studies, describes the contents of a typical work plan, and discusses the major issues to consider when conducting a treatability study. Section 5 discusses sampling and analysis and quality assurance project plans. Section 6 explains how to interpret the data produced from treatability studies and how to determine if further remedy

design testing is justified. Section 7 lists the references.

This guide is one of a series of guidance documents being developed by EPA. This guide, along with guides being developed for other technologies, is a companion document to the generic guide.<sup>(2)</sup> In an effort to avoid redundancy, supporting information in the generic guide and other readily available guidance documents is not repeated in this document.

The document is not intended to serve as a substitute for communication with regulators and/or experts in the field of solvent extraction. This document should never be the sole basis for the selection of solvent extraction as a remediation technology or the exclusion of solvent extraction from consideration.

As treatability study experience is gained, EPA anticipates further comment and possible revisions to the document. For this reason, EPA encourages constructive comments from outside sources. Direct written comments to:

Mr. Michael Gruenfeld  
U.S. Environmental Protection Agency  
Release Control Branch  
Risk Reduction Engineering Laboratory  
2890 Woodbridge Ave.  
Building 10, 2nd Floor  
Edison, NJ 08837  
(908) 321-6625

## SECTION 2

# TECHNOLOGY DESCRIPTION AND PRELIMINARY SCREENING

This section presents a description of various full-scale solvent extraction technologies and a discussion of the information necessary for prescreening the technology before committing to a treatability test program. Subsection 2.1 describes several types of full-scale solvent extraction systems. For the purpose of this document, full-scale is defined as any system which can process greater than one ton per hour and may include some pilot-scale systems. The quality of the data provided by vendors on specific processes has not been determined. Subsection 2.2 discusses the literature and database searches required, the technical assistance available, and the review of field data required to prescreen these technologies. Technology limitations are also reviewed in this subsection.

### 2.1 TECHNOLOGY DESCRIPTION

Solvent extraction is a separation technology which uses a fluid to remove hazardous contaminants from excavated soils, sludges, and sediments and/or contaminated groundwater and surface water. Solvents used are normally organic based fluids not aqueous as is the case with soil washing systems. The solvent is chosen such that the contaminants have a higher affinity for the solvent than for the contaminated material. Solvent extraction does not destroy contaminants; it concentrates them so that they can be recycled or destroyed more cost effectively. When contaminants are not recycled, solvent extraction must be combined with other technologies in a treatment train to destroy the separated, concentrated contaminants. Although solvent extraction has limited application as a treatment technology for inorganic contaminants, this document is focused on extraction of organic contaminants. Nevertheless, solvent extraction may affect inorganic contaminants even when the process is designed to treat organic contaminants.

Solvent extraction processes can be divided into three general types based upon the type of solvent used: standard solvents, near-critical fluids/liquefied gases, and critical solution temperature (CST) solvents. Each of these process types is discussed in the following subsections. Standard solvent processes (subsection 2.1.2) use alkanes, alcohols, ketones, or similar liquid solvents typically used at ambient pressure. Near-critical fluid/liquefied gas processes (subsection 2.1.3) use butane, isobutane, propane, carbon dioxide (CO<sub>2</sub>) or similar gases

which have been liquefied under pressure at or near ambient temperature. Systems involving CST solvents (subsection 2.1.4) use the unique solubility properties of those compounds to extract contaminants at one temperature where the solvent and water are miscible and then separate the concentrated contaminants from the water fraction at another temperature. Solvent is then removed from the contaminants by evaporation. Pretreatment and post-treatment are frequently required for solvent extraction systems. Subsections 2.1.1 and 2.1.5 present a general discussion of various pretreatment and post-treatment needs, respectively.

Solvent extraction shows promise for treating a variety of organic contaminants commonly found at CERCLA sites. The technology has been used as a full-scale remedy at two CERCLA sites: (1) the Treban PCB site in Tulsa, OK and (2) the General Refining site in Garden City, GA. During fiscal year 1989, solvent extraction was selected in combination with other technologies for remediation of five Superfund sites having soils and sediments contaminated with polychlorinated biphenyls (PCBs), polynucleararomatic hydrocarbons (PAHs), pentachlorophenol (PCP), and other organic compounds. These sites are Norwood PCBs, MA; O'Conner, ME; Pinette's Salvage Yard, ME; Ewan Property, NJ; and United Creosoting, TX.<sup>(29)</sup>

Information on the technology applicability, the latest performance data, the status of the technology, and sources for further information is provided in one of a series of engineering bulletins being prepared by EPA Risk Reduction Engineering Laboratory (RREL) in Cincinnati, Ohio.<sup>(25)</sup>

#### 2.1.1 Pretreatment

The preparation of feed materials prior to treatment is an important factor in all extraction processes. The purpose of pretreatment is to ensure that the material is in a physical/chemical condition suitable to the characteristics of the treatment process. Pretreatment strategies depend on whether the feed is primarily solids or liquids. Pretreatment involves physical processing and, in some cases, chemical conditioning after the contaminated materials have been removed from their original location.

Since solvent extraction is an ex situ treatment, contaminated soils and sediments must be either excavated

or dredged. Contaminated liquid wastes, including pumpable sludges, are removed and transported using pumps.

Pretreatment for solid feed material typically involves physical unit operations, such as solid-liquid separation, mixing, screening, wet classification, floatation, and size reduction. These operations are selected and used to optimize performance, protect equipment from damage by debris, and/or maximize the types of equipment which can be utilized. Solid-liquid separation improves the performance of processes using solvents which are hampered by the presence of water. Reducing moisture content can also be accomplished with excavation and air drying of the soil. For continuous processes, mixing with solvent or other liquid may be necessary in order to produce a pumpable slurry. Screening prevents larger debris and rocks from damaging process equipment. Batch processes, unlike most continuous processes, can tolerate coarse solids without damage to equipment. Wet classification and floatation are alternative separation techniques to screening. Size reduction aids extraction by breaking large particles into smaller ones and increasing exposed surface area. This results in higher extraction efficiencies and shorter treatment times. Too much size reduction or an overabundance of fines can cause problems with phase separation of the solvent and treated solids. The decision to use any of these pretreatment operations would depend on the waste characteristics, operating condition (batch versus continuous), and extraction process being used.

For liquid feed material, pretreatment may involve some type of solids removal. This can be accomplished by such methods as filtering, screening, or settling. Depending upon the type of solvent extraction system used, the addition of solvent or water may be needed to make sludges more pumpable.

The use of chemical conditioning agents varies widely and is highly dependent upon treatment equipment, materials-of-construction, natural buffering capacity of the matrix, and chemical properties of the pollutants of concern. Common chemical processes include pH adjustment and chelating agent addition to influence the partitioning of constituents between phases. To protect process equipment and possibly avoid solvent degradation, pH adjustments may be needed.

## 2.1.2 Standard Solvent Extraction Processes

In standard solvent extraction processes, solvents such as alkanes, alcohols, or ketones are used to remove contaminants from excavated soils, sludges, and sediments. Some processes may be applicable to liquid media. The solvents are mixed with the contaminated media (solids, liquids, or both) at essentially standard temperature and pressure. Figure 2-1 is a general schematic of a typical standard solvent extraction process. The system may be operated in either a batch or continuous mode and consists of four steps: (1) extraction, (2) separation, (3) desorption, and (4) solvent recovery.

In the first step, solvent extraction (1), contaminants are extracted from the contaminated media. In this step, the solvent is mixed with the contaminated media for a

specified period of time. The contact time and type of solvent used are contaminant-specific and are typically chosen during treatability studies.

After the appropriate mix time the mixture is allowed to phase separate in the second step, separation (2). This step may not be required. If a solid and liquid phase are formed, the liquid phase is decanted. A soil/sediment phase, which may contain some residual solvent, will be formed if a solid matrix or sludge is being treated. Regardless of the type of solid being treated, a liquid phase containing the solvent, any extracted contaminants, and fine materials will form.

Process water or moisture from the feed either remains in the solid phase or is transported to the solvent phase depending on the process used. In some processes, excess water may be deleterious.

Water is typically removed from the decontaminated phase before the material enters the third step, desorption (3). Residual solvent is removed from the soil/sediment phase by vapor or steam stripping or by indirect heating with hot inert gases and/or steam in the desorption unit. Removed solvent is sent to the extractor. Decontaminated soil/sediment is returned to the site or sent offsite for disposal. Post-treatment of residual solids is addressed in subsection 2.1.5 of this document.

In the final step, solvent recovery (4), solvent is recovered in a distillation system, combined with recovered solvent from step 3, and recycled to the extractor. Still bottoms, which contain concentrated contaminants, are removed from the distillation unit periodically for final treatment or reuse as raw material if of sufficient quality.

While a number of vendors are using systems similar to the system described above, there are also variations. Examples of these variations are evident in the extraction processes described within this section.

A New York University research team, funded by EPA, developed the Low Energy Extraction Process (LEEP<sup>™</sup>) to extract PCBs and other hydrophobic (immiscible in water) organic contaminants from soil, sediment, and sludge. ART International (formerly Applied Remediation Technology) has commercialized the LEEP<sup>™</sup> technology. Excess water is separated from soils and sediments by filtration if required. Hydrophobic contaminants are removed using a hydrophilic solvent contacted in a counter-current leaching unit. The hydrophilic leaching solvent is able to penetrate and remove the water film, which can interfere with the solvent extraction process, from the surface of wet soils and sediments. The water-solvent mixture containing the contaminant is then extracted with a hydrophobic solvent in a countercurrent, liquid-liquid extractor. The contaminant-free hydrophilic leaching solvent is recycled by distillation. Relatively small amounts of energy are used because the selected hydrophilics boil at relatively low temperatures with low latent heat values. Contaminants are concentrated in the hydrophobic solvent which will require additional treatment. Contaminants in the water from the initial solid-water separation step are adsorbed onto a small portion of the cleaned soil. Contaminated soils from the adsorption step are added to the primary feed stream and processed through the solvent extraction system for decontamination.<sup>(6)</sup>

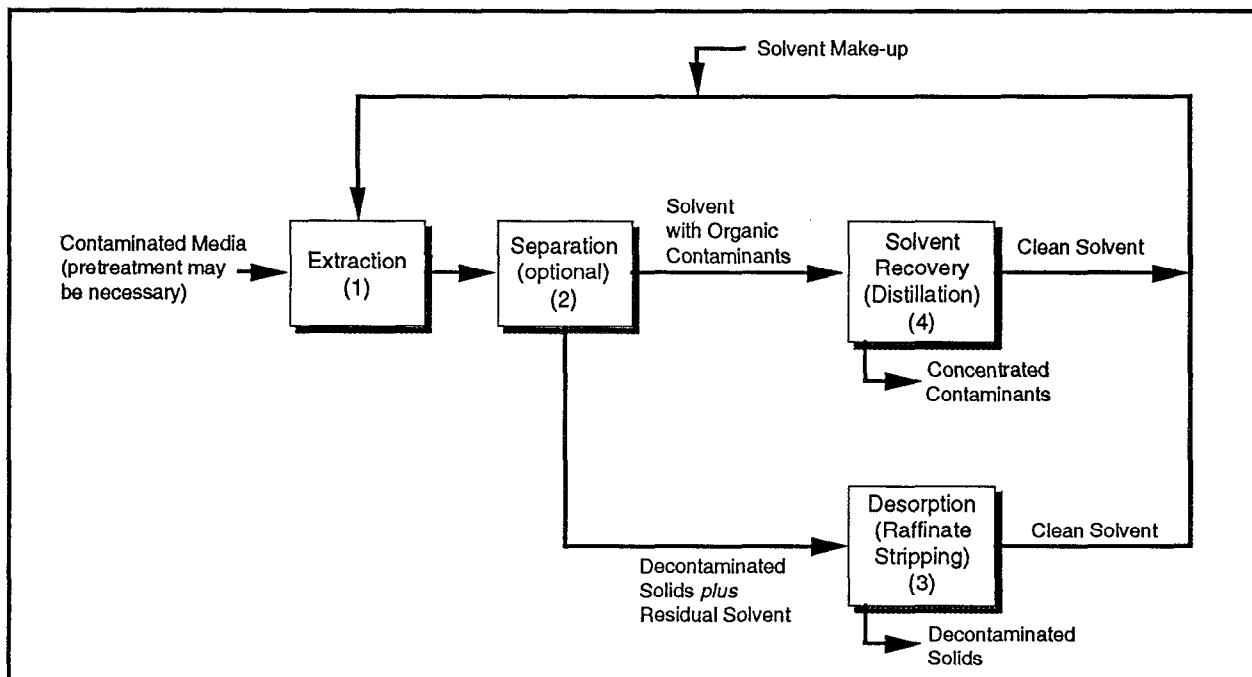


Figure 2-1. General schematic of a standard solvent extraction process.

Nukem Development (formerly ENSR), Houston, Texas, is developing a mobile solvent extraction process to decontaminate soils and sludges without significant pretreatment of the soil/sludge. No addition or removal of water is required. A chemical agent is added with the solvent to neutralize the effects of the moisture present in the soil/sludge. The soil/sludge is mixed with the reagent and solvent and then fed through a series of three to five extraction stages countercurrent to the solvent. The mixture is stripped of residual solvent and transferred to a tank for separation of water from soils.<sup>(13)</sup>

The Sanivan Group, now CET Environmental Services and part of Consolidated Environmental Technologies, has developed two processes. One is a transportable modular solvent extraction process called Extraksol<sup>™</sup>. This batch system involves several steps. In the first step, solid material is loaded into the extraction vessel where it is washed with fresh solvent. Soil-solvent contact is enhanced by slowly rotating the vessel on its axis. After the soil is decontaminated, the solvent is removed and transferred to a storage tank. The contaminated solvents are continuously regenerated by distillation, and the concentrated contaminants are collected in drums for offsite disposal/treatment.<sup>(15)(33)</sup> In the next step, residual solvent in the decontaminated soil is driven off by recirculating hot inert gas within the extraction vessel. The second process is a mobile solvent extraction process called Decontaksolv<sup>™</sup>. It uses an autoclave in a vapor degreasing mode to decontaminate rocks, debris, equipment, and miscellaneous materials found in contaminated sites. The extraction fluid used in this second process is also regenerated by distillation<sup>(6)</sup>.

Terra-Kleen has commercialized the Soil Restoration Unit, a mobile solvent extraction process. The process is

designed for use with a selection of 14 non-toxic solvents. The solvent or solvent combination chosen is governed by the contaminants to be extracted from the soil or debris. The process is typically operated at elevated temperatures. The soil is mixed with the solvent in a counter-current method. The collected solvent is distilled for reuse, while the clean soil/solvent slurry is sent to a drying chamber for removal of the solvent.<sup>(20)(21)</sup>

A laboratory-scale solvent extraction process was used by the Emergencies Engineering Division (EED) of Environment Canada in a joint project with the Groundwater and Soil Remediation Program (GASRep) to compare the effectiveness of two solvents: hexane and natural gas condensate. This batch process had a mixing chamber where contaminated soil and solvent were contacted and allowed to settle. During the comparison, free liquid was decanted. The post-mix slurry was centrifuged, resulting in another decanted liquid stream and a decontaminated moist soil stream. The two decanted liquid streams and make-up solvent were mixed together and distilled to concentrate the contaminants in the bottoms and to recover solvent. The moist soil was dried to remove residual solvent which was sent to the distillation column.<sup>(17)</sup>

Martin Marietta's Soilex process was the result of an effort to remediate PCB-contaminated soil at the Department of Energy's Y-12 plant in Oak Ridge, Tennessee. The pilot plant was operated and evaluated using a 50/50 mixture of kerosene and water. Three extraction stages were used, with soil and water added to the first stage and clean kerosene added to the third stage. The soil-water phase was transferred by gravity from the first to the second stage and then on to the third stage, while kerosene was

transferred by pump countercurrently. Air-driven mixers provided agitation. Kerosene extracted the PCB and oil contaminants in the soil while the water served to break up soil particles. After mixing, the solvent was decanted. The decanted solvent from the first stage, contaminated with PCB and oil, was sent to a packed column distillation system. The processed soil from the third stage, saturated with a significant amount of solvent, was removed from the process.<sup>(19)</sup>

Phønix Miljø, Denmark has developed the Soil Regeneration Plant, a 10 ton/hour transportable solvent extraction process. This process consists of a combined liquid extraction and steam stripping process operating in a closed loop. A series of screw conveyors is used to transfer the contaminated soil through the process. Contaminants are removed from soil in a countercurrent extraction process. A drainage screw separates the soil from the extraction liquid. The extraction liquid is distilled to remove contaminants and is then recycled. The soil is steam heated to remove residual contaminants before exiting the process.<sup>(16)</sup>

The Carver-Greenfield Process has been designed by Dehydro-Tech Corporation, East Hanover, NJ to separate materials into their constituent solid, oil (including oil-soluble substances), and water phases. It is intended mainly for soils and sludges contaminated with oil-soluble hazardous compounds. The technology uses a food-grade carrier oil to extract the oil-soluble contaminants. Pretreatment is necessary to achieve particle sizes of less than 1/4 inch. The carrier oil, with a boiling point of 400 degrees Fahrenheit, is typically mixed with waste sludge or soil, and the mixture is placed in an evaporation system to remove any water. The oil serves to fluidize the mix and maintain a low slurry viscosity to ensure efficient heat transfer, allowing virtually all of the water to evaporate. Oil-soluble contaminants are extracted from the waste by the carrier oil. Volatile compounds present in the waste are also stripped in this step and condensed with the carrier oil or water. After the water is evaporated from the mixture, the resulting dried slurry is sent to a centrifuging section that removes most of the carrier oil and contaminants from the solids. After centrifuging, residual carrier oil is removed from the solids by a process known as "hydroextraction". The carrier oil is recovered by evaporation and steam stripping. The hazardous constituents are removed from the carrier oil by distillation. This stream can be incinerated or reclaimed. In some cases, heavy metals in the solids will be complexed with hydrocarbons and will also be extracted by the carrier oil.

### 2.1.3 Near-Critical Fluid/Liquefied Gas Solvent Extraction Processes

Near-critical fluid/liquefied gas extraction is similar to standard solvent extraction. The difference is that the solvent is near its thermodynamic critical point (the temperature and pressure at which the liquid and vapor phases of the solvent in equilibrium with each other become identical, forming one phase). As a fluid approaches its critical point it increasingly exhibits the diffusivity and viscosity characteristics of a gas, while continuing to exhibit the solvent characteristics of a liquid. Thus a solvent near its critical point can effectively

penetrate a soil matrix with rapid mass transfer and remove pollutants. Near-critical fluid/liquefied gas extraction processes generally operate at elevated pressure. Processes have been designed to handle either solids or liquids. Figure 2-2 is a general schematic of a typical near-critical fluid/liquefied gas extraction process, which is a continuous cycle consisting of four steps: (1) extraction, (2) separation, (3) desorption, and (4) solvent recovery.<sup>(14)</sup>

Contaminated media is pretreated (see subsection 2.1.1), transferred into an extraction vessel, and mechanically mixed with solvent (1). Vigorous mixing is required to thoroughly disperse the hydrophobic solvent into the contaminated media. The extraction step can involve one or more extraction stages where solvent and feed move in countercurrent directions.

The separation step (2) is the part of the process where the separation of the two phases, decontaminated media and contaminated solvent, occurs. The decontaminated media settles to the bottom, and consists of the treated liquid and material fines as well as residual solvent which is vaporized and separated from the treated materials in the desorption step (3). The decontaminated media are subsequently discharged. The vaporized residual solvent is compressed and recycled back to step 1.

The contaminated solvent, which contains the organic contaminants, rises to the top of the separation chamber. The mixture flows to the solvent recovery step (4) where a combination of reduced pressure and additional heat vaporize the solvent and separate it from the organic contaminants. The contaminants are subsequently discharged, and the solvent is recompressed and cycled back to the extraction step.<sup>(23)</sup>

Examples of this type of extraction are the proprietary processes of CF Systems. CF Systems designs include a liquid propane/butane solvent process for treatment of soils and sludges and a liquefied carbon dioxide (CO<sub>2</sub>) gas process for treatment of wastewater. Waste sludges to be treated are pumped as slurries while soils are loaded directly into the extractor. Their liquid propane/butane process consists of a multi-stage mixer settler arrangement. The liquefied CO<sub>2</sub> process has one multi-stage extraction tower.<sup>(23)</sup>

Sierra Environmental Services, Inc. intends to market a liquid/liquid extraction process using liquid butane as the solvent. This process was developed under sponsorship by the Emergencies Engineering Division of Environmental Canada. Tests in both a small, single-stage, bench-scale unit (capacity approximately 0.75 L) and a continuous, counterflow pilot-plant with four actual mixing stages (80 to 100 mL/min. water: 15 to 25 mL/min. butane) have been completed. During this work, a total of 25 different organic pollutants were tested, either singly or in combination with water.<sup>(1)</sup>

The near-critical fluid/liquefied gas extraction solvents discussed thus far are sometimes referred to as near-critical liquids (NCL). Bench scale studies have also investigated the use of supercritical fluids (SCFs). These SCFs are fluids heated and pressurized beyond their critical temperatures and critical pressures. Three SCF approaches are being examined. The first is a two-step process in which an adsorbent such as activated carbon is

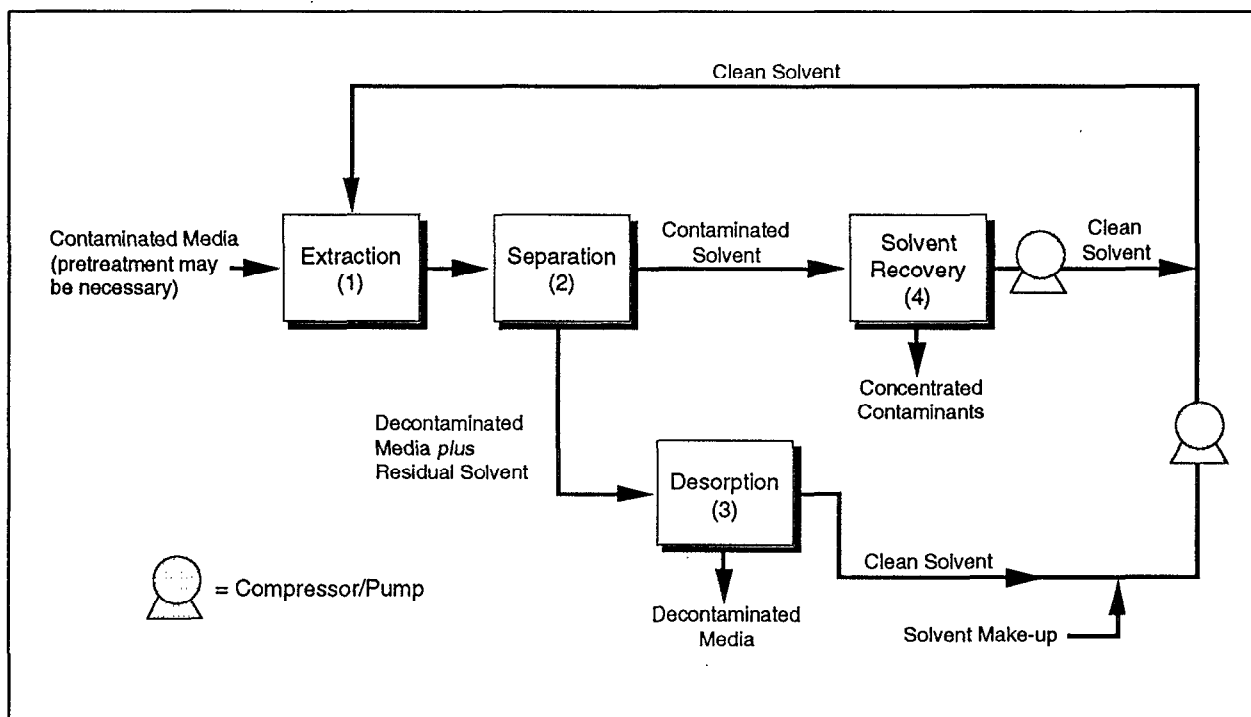


Figure 2-2. General schematic of a near-critical fluid/liquified gas solvent extraction process.

used to concentrate organic contaminants and is then regenerated by extraction with a SCF. The second approach involves the use of supercritical water to simultaneously extract contaminants and oxidize them with the addition of air or pure oxygen. The third approach uses nontoxic SCFs such as CO<sub>2</sub>, hydrocarbons, and freons to remove organic contaminants from water.<sup>(10)</sup>

## 2.1.4 Critical Solution Temperature (CST) Processes

CST processes use extraction solvents in which solubility characteristics can be enhanced by changing the fluid's temperature. For the purpose of this document, CST solvents include those binary (liquid-liquid) systems which exhibit an upper critical solution temperature (sometimes referred to as upper consolute temperature), a lower critical solution temperature (sometimes referred to as lower consolute temperature), or both. For such systems, mutual solubilities of the two liquids increase while approaching the CST. At or beyond the CST the two liquids are completely miscible in each other. Additional information on CST solvents can be obtained from textbooks on liquid-liquid equilibria.<sup>(5)(9)</sup> Figure 2-3 is a general schematic of a typical lower CST solvent extraction process. The process consists of four steps: (1) extraction, (2) separation, (3) desorption, and (4) solvent recovery. Step 4 is complex and involves many unit operations.

During the first step, pretreated contaminated media (soil or sludge) enters the extractor (1) and is contacted with a CST solvent which is cooled or heated until complete miscibility in water is exhibited. The water and contaminants within the soil/sludge dissolve into the

cooled or heated CST solvent, forming a homogeneous liquid. Since only one liquid layer is formed, the solids can be easily removed from the slurry by physical means such as filtering, settling, and/or centrifuging in the second step, separation (2).<sup>(39)</sup>

In the third step, desorption (3), residual solvent is recovered from the solids. This is normally accomplished by drying the solids with direct heat and condensing the solvent vapor driven off. Solvent vapor from the dryer is combined with solvent vapor from the strippers discussed in step 4.

Solvent recovery (4) is the fourth process step. The temperature of the liquid portion from the extraction step (the solids were previously removed) is modified so that the solvent is immiscible in water. Depending on the type of solvent used, the temperature may be raised or lowered to form a binary liquid system. The contaminated solvent-water mixture separates into two distinct layers in the decanter. One layer containing mostly solvent along with the extracted contaminants, the other containing mostly water. The solvent fraction is steam stripped to recover a solvent-water mixture or azeotrope and a concentrated contaminant product. The water fraction is steam stripped also, yielding a solvent-water mixture or azeotrope and a treated water product. The recovered solvent fractions are combined, condensed, and decanted once more, if required. Solvent from this final decanting is used in the extraction process again. Water from this final decanting is recycled to the water fraction steam stripper.

Resources Conservation Company (RCC) has a patented CST extraction process called B.E.S.T.<sup>TM</sup> which uses triethylamine as the extraction solvent. Solvent and water



cooled. It is similar to the generalized description with the exception that a second extraction step takes place with the solids from the first centrifuge. Feed pretreatment, consisting of pH adjustment, is needed in order to keep the aliphatic amine solvent stable.<sup>(28)(39)</sup>

## 2.1.5 Post-Treatment

Solvent extraction is not a stand-alone technology. Typically, the concentrated contaminants, the fine soils (silts and clays), and any separated water are subject to further specific treatment and disposal techniques, as appropriate, to complete the cleanup. Sidestreams generated during treatment, such as spent solvent, spent activated carbon, air emissions, etc., must also be treated. Solvent extraction systems are generally designed to operate without air emissions. Nevertheless, volatile air emissions requiring treatment could occur during waste preparation. The EPA document entitled "Technology Screening Guide for Treatment of CERCLA Soils and Sludges" contains a description of potential treatment technologies which may be used as post-treatments for residual solids.<sup>(36)</sup>

The concentrated contaminants, which are usually the residual from solvent recovery, may or may not meet the specifications required for disposal, recycle, or reuse. If

not, further treatment with another technology is necessary.

Treated soil or sludge will, at minimum, have traces of extraction solvent present. If little or no effort is made to recover and recycle the extraction solvent during processing, the amount of residual extraction solvent could be significant.

Typically, extraction solvents used in commercially available systems either volatilize quickly or are biodegradable. Ambient air monitoring can be employed to determine if the volatilizing solvents present a problem. Depending on the system, clean soil and solids from treated sludge or sediments may need dewatering in order to form a dry solid and a separate water stream. Even though solvent extraction systems designed for organic contaminant removal may have some effect on metals or other inorganic contaminants, such metals or other inorganics are frequently not extracted, and their presence may indicate the need for additional treatment of the cleaned solids by another technique. Some inorganics may be removed in the fine silt fraction which is removed with the silt bottoms. Therefore, further treatment of the total waste volume may not be necessary.

Residual water from decantation, dewatering or stripping is normally treated using standard wastewater treatment

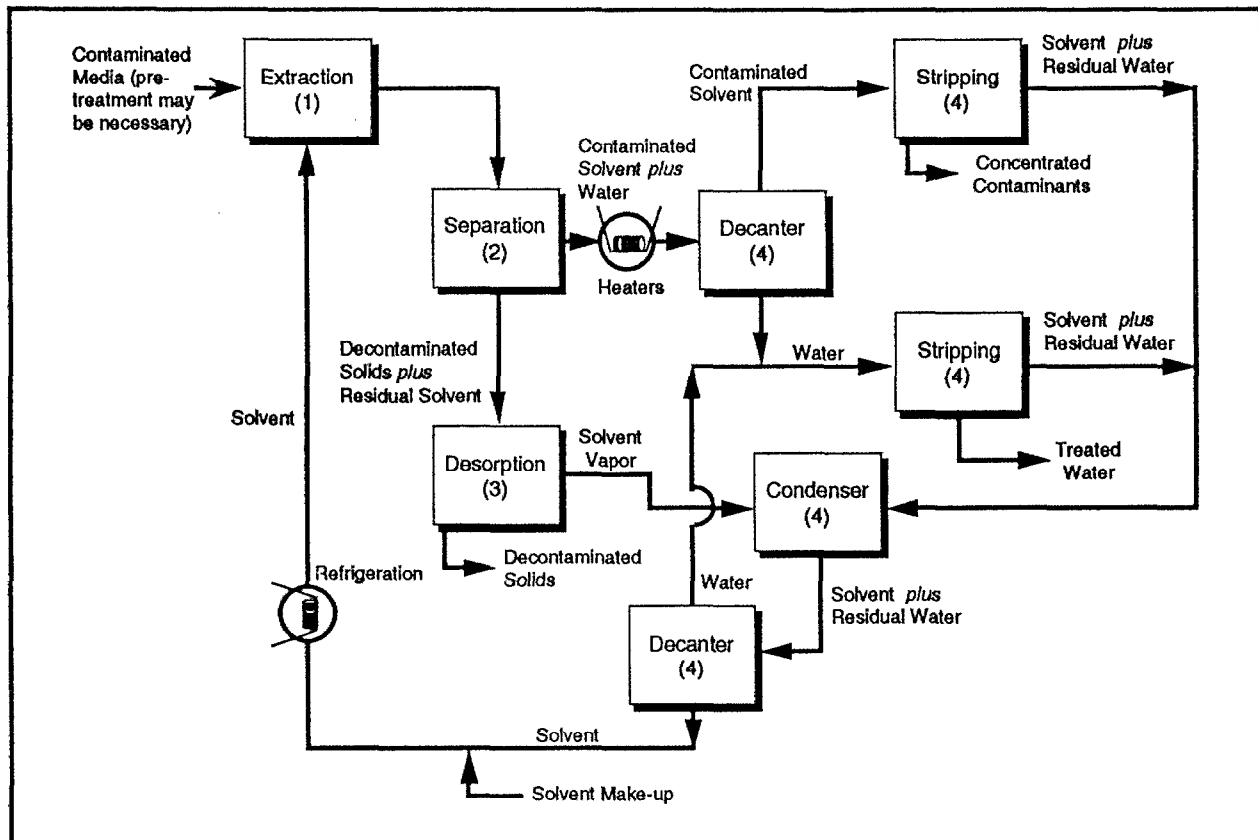


Figure 2-3. General schematic of a CST solvent extraction process.

practices. Sludges generated during water treatment may need subsequent treatment.

## 2.2 PRELIMINARY SCREENING AND TECHNOLOGY LIMITATIONS

The determination of the need for and the appropriate level of treatability studies is dependent on available literature, expert technical judgment, and site-specific factors. The first two elements—the literature search and expert consultation—are critical factors in determining if adequate data are available or whether a treatability study is needed to provide those data.

### 2.2.1 Literature/Database Review

Several reports and electronic databases exist which should be consulted to assist in planning and conducting treatability studies and to help prescreen solvent extraction for use at a specific site. Existing reports include:

- Guide for Conducting Treatability Studies Under CERCLA, Interim Final. U.S. Environmental Protection Agency, Office of Research and Development and Office of Emergency and Remedial Response, Washington, D.C. EPA/540/2-89/058, December 1989.
- Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, D.C. EPA/540/G-89/004, October 1988.
- Superfund Treatability Clearinghouse Abstracts. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, D.C. EPA/540/2-89/001, March 1989.
- The Superfund Innovative Technology Evaluation Program: Technology Profiles. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response and Office of Research and Development, Washington, D.C. EPA/540/5-89/003, November 1988.
- Summary of Treatment Technology Effectiveness for Contaminated Soil. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, D.C. EPA/540/8-89/053, 1989.
- Technology Screening Guide for Treatment of CERCLA Soils and Sludges. U.S. Environmental Protection Agency. EPA/540/2-88/004, September 1988.

Currently, the Risk Reduction Engineering Laboratory (RREL) in Cincinnati is expanding the RREL Treatability Data Base. This expanded database will contain data from soil treatability studies. A repository for the treatability study reports will be maintained at RREL in Cincinnati. The contact for this database is Glenn Shaul (513) 569-7408.

The Office of Solid Waste and Emergency Response (OSWER) maintains the Cleanup Information (CLU-IN) Bulletin Board System for communicating ideas, disseminating information, and serving as a gateway for

other OSW electronic databases. Currently, the CLU-IN Bulletin Board has eight different components, including news and mail services, and conferences and publications on specific technical areas. The contact is Dan Powell (703) 308-8827.

ORD headquarters maintains the Alternative Treatment Technology Information Center (ATTIC), which is a compendium of information from many available data bases. Data relevant to the use of treatment technologies in Superfund actions are collected and stored in ATTIC. ATTIC searches other information systems and databases and integrates the information into a response. It also includes a pointer system that refers the user to individual experts in EPA. The system currently encompasses technical summaries for SITE program abstracts, treatment technology demonstration projects, industrial project results, and international program data. Contact the ATTIC System Operator at (301) 670-6294, access the database from a modem by calling (301) 670-3808, or call the EPA contact at (408) 321-4380.

Finally, the RREL Technical Support Branch is supporting a variety of treatability-related activities, including development of this guide and other technology-specific guidance documents, preparation of engineering bulletins, and compilation of a list of vendors who perform treatability studies.

### 2.2.2 Technical Assistance

Technical assistance can be obtained from the Technical Support Project (TSP) team which is made up of a number of Technical Support Centers. It is a joint service of OSWER, ORD, and the Regions. The TSP offers direct site-specific technical assistance to OSCs and RPMs and develops technology workshops, issue papers, and other information for Regional staff. The TSP:

- Reviews contractor work plans, evaluates remedial alternatives, reviews RI/FS, assists in selection and design of final remedy
- Offers modeling assistance and data analysis and interpretation
- Assists in developing and evaluating sampling plans
- Conducts field studies (soil gas, hydrogeology, site characterization)
- Develops technical workshops and training, issue papers on groundwater topics and generic protocols
- Assists in performance of treatability studies.

As part of the TSP, the Engineering Technical Support Center (ETSC) provides technical information and advice related to treatability studies. The ETSC is sponsored by OSWER but operated by RREL. The Center handles site-specific remediation engineering problems. Access to this support Center must be obtained through the EPA project manager.

RREL offers expertise in contaminant source control structures; materials handling and decontamination; treatment of soils, sludges and sediments; and treatment of aqueous and organic liquids. The following are examples of the technical assistance that can be obtained

**Table 2-1. Major Site Characterization Tests**

Parameter	Description of Test	Method	Purpose and Comments	Application of Data	Ref.
<u><b>Chemical</b></u>					
Organics	Varied	Varied (see SW-846 or other appropriate methods)	To determine concentration of target or interfering constituents, pretreatment needs, extraction medium.	Remedy screening	37
Total organic carbon (TOC) or	Combustion	Method 9060	To determine the presence of organic matter, adsorption characteristics of soil.	Remedy selection	37
Total recoverable petroleum hydrocarbon	Infrared Spectrophotometer	Method 418.1	To determine the presence of organic matter, adsorption characteristics of soil.	Remedy selection	30
<u><b>Physical</b></u>					
Grain size analysis/ particle size distribution	Sieve screening using a variety of screen sizes	ASTM D422	To determine volume reduction potential, pretreatment needs, solid/liquid separability.	Remedy screening	3
Moisture content	Drying oven at 110°C In situ, nuclear method	ASTM D2216 ASTM D3017	To determine pretreatment needs. Water may impede some extraction processes.	Remedy selection	3
Bulk density or	Drive cylinder method Sound-cone method	ASTM D2937 ASTM D1556	To determine throughput capacity in terms of yd <sup>3</sup> or tons per hour.	Remedy screening	3
Specific gravity	Hydrometer Pycnometer Pycnometer	ASTM D891A ASTM D891B ASTM D854	To determine throughput capacity in terms of yd <sup>3</sup> or tons per hour.	Remedy screening	3

through the ETSC:

- Review of the treatability aspects of RI/FS
- Review of RI/FS treatability study Work Plans and final reports
- Oversight of RI/FS treatability studies
- Definition of alternative remedies
- Assistance with studies of innovative technologies
- Assistance in full-scale design and start-up.

For further information on the TSP, contact:

Risk Reduction Engineering Laboratory,  
Cincinnati, OH  
Contact: Ben Blaney  
(513) 569-7406

## 2.2.3 Prescreening Characteristics

Prescreening activities for the solvent extraction treatability testing include interpreting any available site-related field measurement data. The purpose of prescreening is to gain enough information to eliminate from further consideration technologies which have little chance of achieving the cleanup goals.

Table 2-1 lists major site characterization parameters that may be measured or available before designing treatability tests. The "Application of Data" column indicates the tier in which the data is initially used. The most important prescreening parameters are the contaminant profile and concentration of contaminants. Tests for total organic carbon (TOC) and total recoverable petroleum hydrocarbons give an estimate of equilibrium partitioning and contaminant transport between soil and water and may be useful when applying results to other sites with different organic carbon values. Particle size distribution and moisture content are useful for evaluating materials handling and pretreatment processes. Bulk density or specific gravity is important for estimating throughput capacity.

Data on other, less important parameters such as pH, temperature, chemical oxygen demand (COD), and contaminant toxicity may also be collected and analyzed. The matrix pH is especially important to processes which utilize aliphatic amines. This is because the aliphatic amines cannot exist in solvent form at pH lower than 10.<sup>(28)</sup> Feed temperature affects the near-critical fluid/liquefied gas process because below 60°F, hydrates may form and inhibit extraction.<sup>(23)</sup> Moisture content is necessary to convert from wet-weight based analytical results to dry-weight based results to facilitate the calculation of the material balance and to determine the extent of water removal or addition required. Chemical oxygen demand (COD) is a measure of the oxygen required to fully oxidize all organic materials present. The Toxicity Characteristic Leaching Procedure (TCLP) test determines the impact of the treatment on leachability of organic and inorganic contaminants which will affect the final disposal of the wastes. Some parameters may or may not be applicable to specific types of solvent extraction processes.

If contamination exists in different soil strata or in different media, a characterization profile should be developed for

each soil type or media. Available chemical and physical data (including contaminant concentration averages and ranges) and the volumes of the contaminated soil requiring treatment should be identified. For "hot spots", separate characterizations should be done so they can be properly addressed in the treatability tests. Solvent extraction may be applicable to some parts of a site, but not to other parts.

Characterization test results should be broadly representative of the contaminant profile of the site. Grab samples taken from the site ground surface may represent only a small percentage of the contaminated soils requiring remediation.

Contaminant characteristics such as those listed below may be important for the design of remedy screening studies and related residuals treatment systems.

- Composition
- Vapor pressure
- Solubility in specified solvent(s)
- Henry's Law constant
- Partition coefficient
- Boiling point

Matrix characteristics such as the bulk density of solids or the specific gravity and viscosity of sludges and liquids may also be important for the design of treatability studies (e.g., separation, transfer, and mixing techniques).

The need for a treatability study is determined near the beginning of the RI/FS when a literature survey of remedial technologies is performed. Remedial technologies are identified based on compatibility with the type of contaminants present at the site, the waste media (soil, water, etc.), and the anticipated cleanup objectives. Remedial technologies are prescreened for effectiveness, implementability, and cost. The prescreening is done using available technical literature, databases, and manufacturer's information. Based upon this initial technology prescreening, solvent extraction may be one of several candidate remedial technologies selected for further investigation or eliminated during the remedial investigation/feasibility study. See the generic guide for more specific details on screening of treatment technologies and on determining the need and type of treatability tests which may be required for evaluating treatment technology alternatives.<sup>(27)</sup>

## 2.2.4 Solvent Extraction Limitations

Solvent extraction limitations may be defined as characteristics that hinder cost-effective treatment of the contaminated media with specific processes. The limitation may be due to the contaminant (incompatibility with the selected solvents or complex mix of contaminants), the process, or the media. Several extraction stages may be required in some cases to meet the site cleanup goals. Difficulties may be encountered in recycling spent solvents. Hydrophobic and hydrophilic contaminants may be difficult to extract with the same solvent. The contaminated media might require substantial pretreatment.

Complex mixtures of contaminants in the waste media, such as a mixture of metals, non-volatile organics, semi-volatile organics, etc., may make the design or selection of a suitable solvent extraction system that will remove all the different types of contaminants difficult. Organically bound metals can co-extract with the target organic pollutants and restrict disposal and recycle options. The presence of emulsifiers and detergents can adversely affect the extraction performance by competing with the extraction solvent for retention of the organic pollutants. Emulsifiers and detergents can also lead to foaming, which hinders separation and settling characteristics and reduces material throughput.<sup>(25)</sup> Methods are available for breaking foams and emulsions, and these have often been used to facilitate extraction processes. Sequential extraction steps, using different solvents, may be needed. Frequent changes in the contaminant type and concentration in the feed material can disrupt the efficiency of the process. To accommodate such changes in the feed, modifications to the solvent mix and the operating settings may be required. Alternatively, additional feedstock preparation steps may be necessary. High moisture content can interfere with the efficiency of some solvents (i.e. methanol), limiting the application of certain solvent extraction processes.

Advantages and disadvantages exist between the various types of solvent extraction processes described in this section. The primary differences include the following: ability to handle fines or high clay content, ability to handle a wide variety of organic contaminants, the ease of phase separation after extraction, and the energy requirements.

The presence of fines or high clay content may present problems with standard solvent extraction processes. If the contaminants are adsorbed strongly to the waste matrix, the solvent may not be able to remove them. Standard solvent processes are able to use numerous solvents and combinations of solvents and therefore can be used for many different organic contaminants. Phase separation after extraction can be poor at times and may

require mechanical devices such as centrifuges or filters. The energy requirements for separation are usually small for standard solvent processes.

Near-critical fluid/liquefied gas processes are generally better able to deal with fines or high clay content than other solvent types because of the low viscosity and density of the solvent which allows penetration into the clay, and may facilitate solvent/solids separation. Although a large number of near-critical fluid and liquefied gas solvents have been tested, practical, environmental applications have been limited to a few solvents, with or without cosolvents. The primary use of near-critical fluid/liquefied gas processes has been to extract oily contaminants and solvents such as chlorinated hydrocarbons and ketones. The primary limitation which is unique to near-critical fluid/liquefied gas processes is that, because the solvents tend to be nonpolar, very polar organics and high molecular weight contaminants may be difficult to extract. Phase separation after extraction for near-critical fluid/liquefied gas processes is excellent. Once the pressure is reduced, the density difference between the solvent and extracted waste is very high. The energy requirements are typically low for the near-critical fluid/liquefied gas solvent processes. The energy requirements for these processes can be substantially less than for super-critical fluid processes.

The ability of CST solvent processes to handle fines or high clay content may be somewhat superior to that of standard solvent processes. This is because of the ease of phase separation normally experienced with CST solvents. CST solvent processes have limited choices for solvents which can be practically applied, and therefore may not be applicable to some contaminants. The ease of phase separation probably is somewhere in between that of standard solvent and near-critical fluid/liquefied gas processes. Energy requirements are normally higher than with standard solvent processes because of the need for both refrigeration and heating of the solvent.

## **SECTION 3**

# **THE USE OF TREATABILITY STUDIES IN REMEDY EVALUATION**

This section presents an overview of the use of treatability tests in confirming the selection of solvent extraction as the technology remedy under CERCLA. It also provides a decision tree that defines the tiered approach to the overall treatability study program with examples of the application of treatability studies to the RI/FS and remedy selection process. Subsection 3.1 presents an overview of the general process of conducting treatability tests. Subsection 3.2 defines the tiered approach to conducting treatability studies and the applicability of each tier of testing, based on the information obtained, to assess, evaluate, and confirm solvent extraction technology as the selected remedy.

### **3.1 PROCESS OF TREATABILITY TESTING IN SELECTING A REMEDY**

Treatability studies should be performed in a systematic fashion to ensure that the data generated can support the remedy evaluation process. This section describes a general approach that should be followed by RPMs, PRPs, and contractors during all levels of treatability testing. This approach includes:

- Establishing data quality objectives
- Selecting a contracting mechanism
- Issuing the Work Assignment
- Preparing the Work Plan
- Preparing the Sampling and Analysis Plan
- Preparing the Health and Safety Plan
- Conducting community relations activities
- Complying with regulatory requirements
- Executing the study
- Analyzing and interpreting the data
- Reporting the results.

These elements are described in detail in the generic guide.<sup>(27)</sup> That document gives information applicable to all treatability studies. It also presents information specific to remedy screening, remedy selection testing, and remedy design testing.

Treatability studies for a particular site will often entail multiple tiers of testing. Duplication of effort can be avoided by recognizing this possibility in the early planning phases of the project. The Work Assignment, Work Plan, and other supporting documents should include all anticipated activities.

There are three levels or tiers of treatability studies: remedy screening, remedy selection, and remedy design. Some or all of the levels may be needed on a case-by-case basis. The need for and the level of treatability testing required are management decisions in which the time and cost necessary to perform the testing are balanced against the risks inherent in the decision (e.g., selection of an inappropriate treatment alternative). These decisions are based on the quantity and quality of data available and on other decision factors (e.g., state and community acceptance of the remedy and new site data). The flow diagram for the tiered approach in Figure 3-1 traces the stepwise review of study data and the decision points and factors to be considered.

Technologies generally are evaluated first at the remedy screening level and progress through the remedy selection to the remedy design tier. A technology may enter the selection process, however, at whatever level is appropriate based on available data on the technology and site-specific factors. For example, a technology that has been successfully applied at a site with similar conditions and contaminants may not require remedy screening to determine whether it has the potential to work. Rather, it may go directly to remedy selection testing to verify that performance standards can be met. Treatability studies, at some level, will normally be needed even if previous studies or actual implementation have encompassed similar site-specific conditions to assure that the site target cleanup goals are going to be achieved. Figure 3-2 shows the relationship of the three levels of treatability study to each other and to the RI/FS process.

### **3.2 APPLICATION OF TREATABILITY TESTS**

Before conducting treatability studies, the objectives of each tier of testing must be established. Solvent extraction treatability study objectives are based upon the specific needs of the RI/FS. There are nine evaluation criteria specified in the document, Guidance for Conducting Remedial Investigations and Feasibility Studies Under

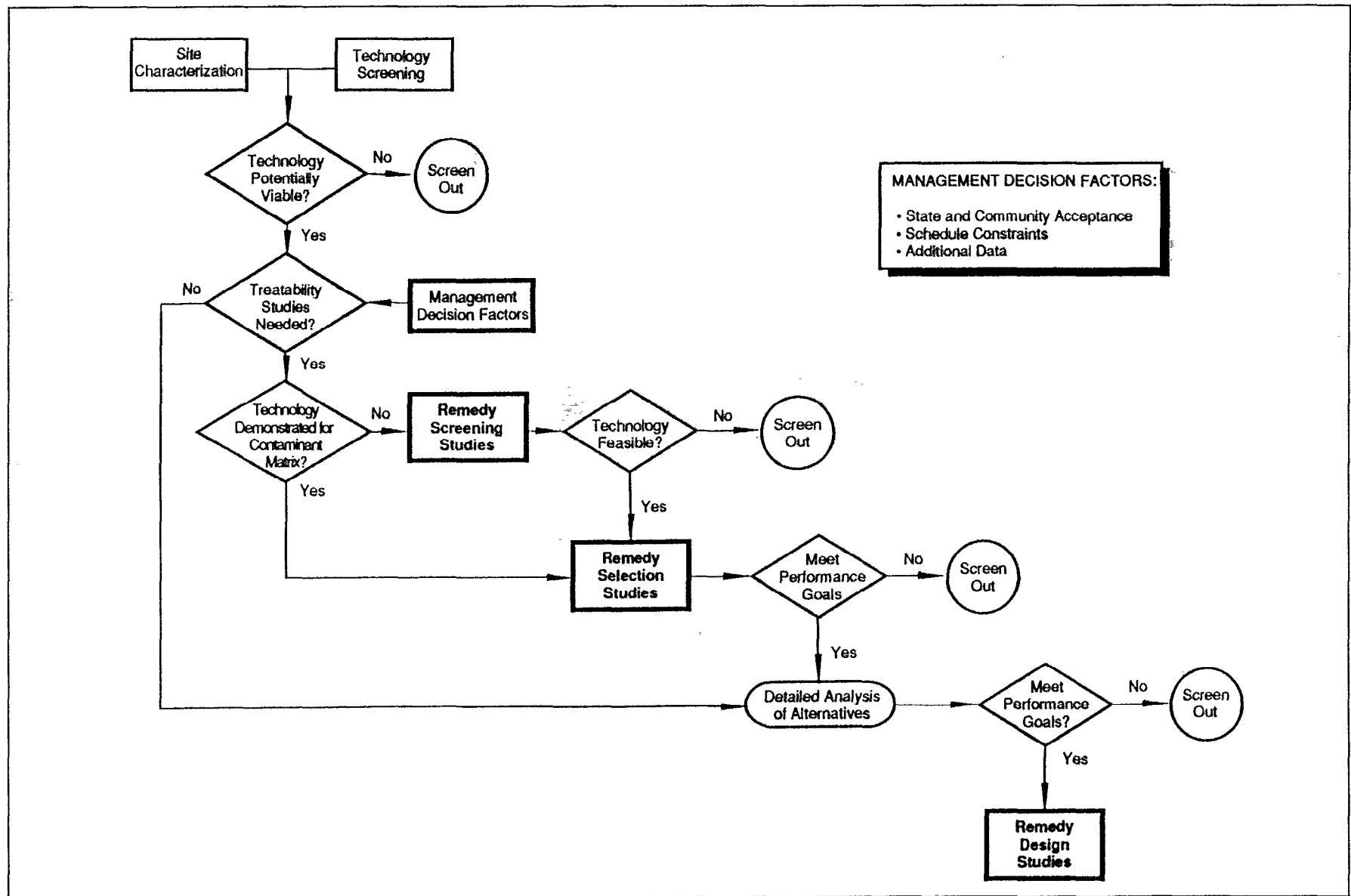


Figure 3-1. Flow diagram of the tiered approach.

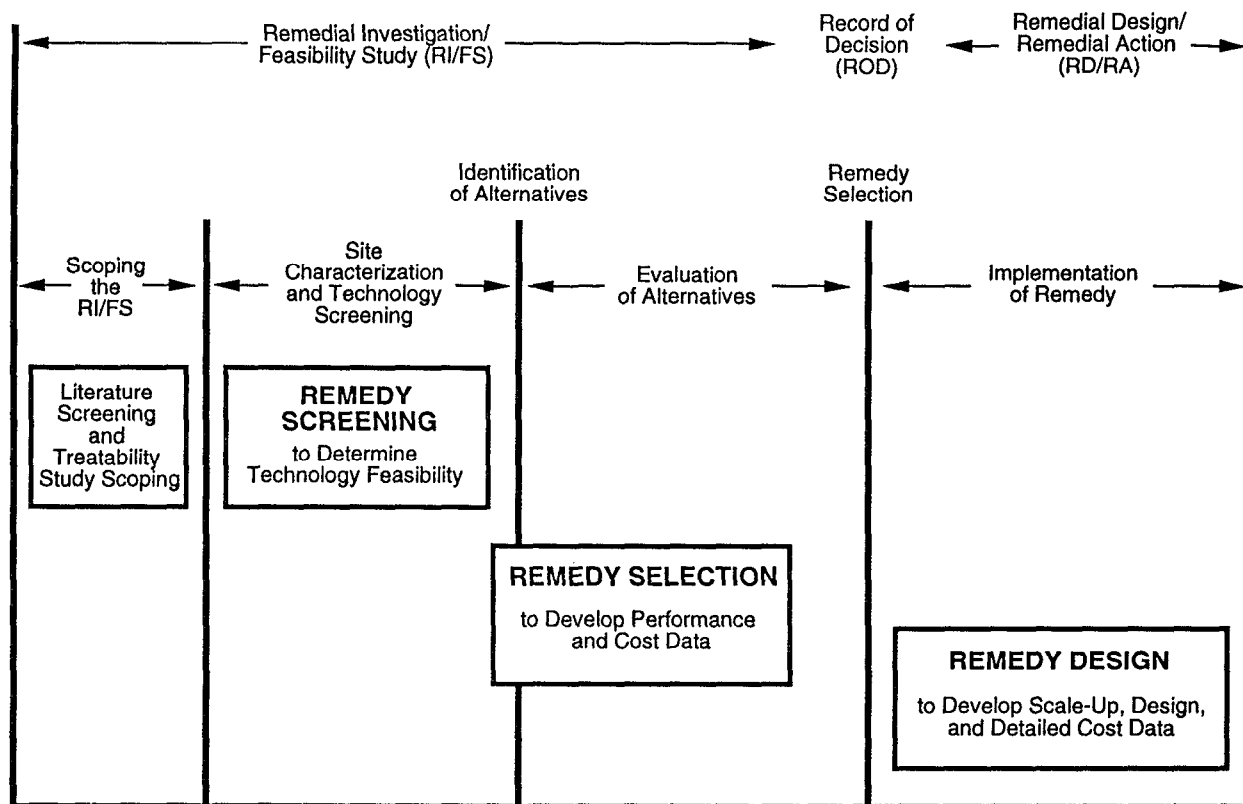


Figure 3-2. The role of treatability studies in the RI/FS and RD/RA process.

CERCLA (Interim Final);<sup>(26)</sup> the treatability studies provide data for up to seven of these criteria. These seven criteria are:

- Overall protection of human health and environment
- Compliance with applicable or relevant and appropriate requirements (ARARs)
- Reduction of toxicity, mobility, or volume through treatment
- Short-term effectiveness
- Implementability
- Long-term effectiveness and permanence
- Cost.

The first four of these evaluation criteria deal with the degree of contaminant reduction achieved by the solvent extraction process. What will be the remaining contaminant concentrations? Will the residual contaminant levels be sufficiently low to meet the established ARARs and the risk-based contaminant cleanup levels? What are the contaminant concentration and physical and chemical differences between the untreated and the treated solids fractions (e.g., has contaminant toxicity, mobility, and volume been reduced)? The fourth criterion, short-term effectiveness, also addresses the effects of the treatment technology

during construction and implementation of a remedy. This evaluation is concerned not only with contaminant concentration and toxicity, but also with the potential for exposure to solvents or solvent vapors which may be harmful.

The implementability assessment evaluates the technical and administrative feasibility of the technology and the availability of required goods and services. The following questions must be answered in order to address the implementability of solvent extraction:

- Will solvent residuals in soil and water make residuals treatment and disposal difficult?
- What are the characteristics and the volume of the residuals that will be produced?
- Are the process equipment and solvent readily available?
- Can the solvent be economically recovered and recycled?
- What are the necessary pretreatment steps (specific to the process equipment and solvent)?
- Will the solvent extraction system chemicals react with the solutes?

Normally, the required equipment and extracting solvents are available. However, alterations to process design may be necessary on a site-by-site basis to accommodate



different media and contaminants. Contaminants can be treated onsite with mobile or portable units (modular components constructed onsite) or removed to an offsite facility. Residuals from the solvent extraction process require additional treatment. The implementability assessment must include these additional treatments. The ability to recover and recycle solvents is generally critical to the implementability of solvent extraction.

Long-term effectiveness assesses how effective treatment technologies are in maintaining protection of human health and the environment after response objectives have been met. The magnitude of any residual risk and the adequacy and reliability of controls must be evaluated. Residual risk, as applied to solvent extraction, assesses the risks associated with the treatment residuals at the conclusion of all remedial activities. Analysis of residual risk from sidestream and other treatment train processes should be included in this step. An evaluation of the reliability of treatment process controls assesses the adequacy and suitability of any long-term controls (such as site access restrictions and deed limitations on land use) that are necessary to manage treatment residuals at the site. Such assessments are usually beyond the scope of a remedy selection treatability study, but may be addressed conceptually based on remedy selection results. Performance goals must consider the existing site contaminant levels and relative cleanup goals for soils, sludges, and water at the site. In previous years, cleanup goals often reflected background site conditions. Attaining background cleanup levels through treatment has proved impractical in many situations. The present trend is toward the development of site-specific cleanup target levels that are risk-based rather than background-based.

The final EPA evaluation criterion which can specifically be addressed during a treatability study is cost. The solvent extraction process transfers contaminants to and concentrates them in the solvent. The solvent is typically reclaimed, leaving behind a concentrated waste in the still bottoms. The disposal and/or treatment cost for concentrated waste is less than that for unconcentrated waste. Normally, the treated solid and/or liquid phase has a low contaminant concentration. Because the contaminant concentration is low, further treatment may not be necessary and disposal costs are small. Air emissions are typically minor. The cost savings, in terms of disposal and/or treatment, realized by separating and concentrating contaminants and by reducing the contaminant concentration in the solid and/or liquid phase should cover the cost of treatment by solvent extraction.

Remedy selection treatability studies can provide data to estimate the following important cost factors:

- The volume and characteristics of residual wastewater and sludge which require treatment or disposal.
- The degree to which process modifications can enhance the efficiency of the process.
- The degree to which the solvent and/or contaminant can be recovered and recycled.
- The solvent-to-feed ratio.
- The number of extraction stages necessary.

The first two factors provide information about the costs of downstream treatments by determining the amount and character of the contaminated residuals. The last four factors help estimate the costs of equipment, supplies, and utilities directly associated with the specific solvent extraction system.

Treatability tests do not directly relate to the final two criteria, state and community acceptance, because these criteria reflect the apparent preferences or concerns about alternative technologies of the state and the community. A viable remediation technology may be eliminated for consideration if the state or community objects to its use. However, treatability studies may provide data that can address state and community concerns and in some cases change their preferences.

### 3.2.1 Remedy Screening

Remedy screening is the first level of testing. It is used to establish the ability of a technology to treat a waste. These studies are generally low cost (e.g., < \$30,000) and usually require one or more days to complete the testing. Additional time must be allowed for project planning, chemical analyses, interpretation of test data, and report writing. Only limited quality control is required for remedy screening studies. They yield data indicating a technology's potential to meet performance goals. Remedy screening tests can identify operating standards for investigation during remedy selection or remedy design testing. They generate little, if any, design or cost data and should not be used as the sole basis for selection of a remedy.

Solvent extraction remedy screening treatability studies are occasionally skipped, if there is enough information about the physical and chemical characteristics of contaminant and media to allow an expert to evaluate the potential success of solvent extraction at a site. In such cases, remedy selection tests are normally the first level of treatability study executed. When remedy screening studies are performed, certain steps, such as solvent recovery, may be skipped if they are based on existing technology. When performed, remedy screening tests are performed in laboratory-scale extraction equipment. These tests are generic and can be performed at any laboratory with the proper equipment and qualified personnel.

### 3.2.2 Remedy Selection

Remedy selection testing is the second level of testing. Remedy selection tests identify the technology's performance for a site. These studies generally have a moderate cost (e.g., \$20,000 to \$120,000) and require several months or more to plan, obtain samples, and execute. Remedy selection tests yield data that verify that the technology can meet expected cleanup goals, provide information in support of the detailed analysis of alternatives (i.e., seven of the nine evaluation criteria), and give indications of optimal operating conditions.

The remedy selection tier of solvent extraction testing consists of either bench-scale tests and/or pilot tests. Typically, these tests are vendor-specific. Sufficient experimental controls are needed such that a quantitative

material balance can be achieved. The key question to be answered during remedy selection testing is whether the treated media will meet the cleanup goals for the site. The exact removal efficiency or acceptable residual contaminant level specified as the goal for the remedy selection test is site-specific. Typically, a remedy design study would follow a successful remedy selection study.

### 3.2.3 Remedy Design

Remedy design testing is the third level of testing. In this tier, pilot tests provide quantitative performance, cost, and design information for remediating an operable unit. This testing also produces the remaining data required to optimize performance. These studies are of moderate to high cost (e.g., \$100,000 to \$500,000) and require several months to complete the testing. As with the other tiers, planning, analysis, and report writing will add to the duration of the study. For complex sites (e.g., sites with different types or concentrations of contaminants in different media such as soil, sludges, and water), longer testing periods may be required, and costs can be higher. Remedy design tests yield data that verify performance to a higher degree than the remedy selection and provide detailed design information. They are performed during the remedy design of a site cleanup after the ROD and

evaluation of alternatives.

Remedy design tests usually consist of bringing a mobile treatment unit onto the site, or constructing a small-scale unit for non-mobile technologies. Permit waivers may be available for offsite treatability studies under certain conditions. For most materials, a permit exclusion is available provided the quantity of material being sent offsite is 4,000 kg or less. The objective of this tier of testing is to confirm the cleanup levels and treatment times specified in the Work Plan (see subsection 4.1.1). This is best achieved by operating a field unit under conditions similar to those expected in the full-scale remediation project.

Data obtained from the remedy design tests are used to:

- Design the full-scale unit
- Confirm the feasibility of solvent extraction based on target cleanup goals
- Refine Cleanup time estimates
- Refine cost predictions

Given the lack of full-scale experience with solvent extraction, remedy design testing will generally be necessary before full-scale implementation.

## SECTION 4

# TREATABILITY STUDY WORK PLAN

Section 4 of this document is written assuming that a Remedial Project Manager is requesting treatability studies through a work assignment/work plan mechanism. Although the discussion focuses on this mechanism, it would also apply to situations where other contracting mechanisms are used.

This chapter focuses on specific elements of the Work Plan for solvent extraction treatability studies. These include test objectives, experimental design and procedures, equipment and materials, reports, schedule, management and staffing, and budget. These elements are described in subsections 4.1 through 4.9. Complementing the above subsections are section 5, Sampling and Analysis Plan and Quality Assurance Project Plan, and section 6, Treatability Data Interpretation, which address the sampling data analysis elements of the Work Plan in greater detail. Table 4-1 lists all of the Work Plan elements.

**Table 4-1. Suggested Organization of Solvent Extraction Treatability Study Work Plan**

No.	Work Plan Elements	Sub-section
1.	Project Description	
2.	Remedial Technology Description	
3.	Test Goals	4.1
4.	Experimental Design and Procedures	4.2
5.	Equipment and Materials	4.3
6.	Sampling and Analysis	4.4
7.	Data Management	
8.	Data Analysis and Interpretation	4.5
9.	Health and Safety	
10.	Residuals Management	
11.	Community Relations	
12.	Reports	4.6
13.	Schedule	4.7
14.	Management and Staffing	4.8
15.	Budget	4.9

Carefully planned treatability studies are necessary to ensure that the data generated are useful for evaluating the validity or performance of a technology. The Work Plan, prepared by the contractor when the Work Assignment is in place, sets forth the contractor's proposed technical approach for completing the tasks outlined in the Work Assignment. It assigns responsibilities and establishes the project schedule and costs. The Work Plan must be approved by the RPM before initiating subsequent tasks. For more information on each of these sections, refer to the generic guide.<sup>(27)</sup>

### 4.1 TEST GOALS

Setting goals for the treatability study is critical to the ultimate utility of the data generated. Objectives must be defined before starting the treatability study. Each tier of the treatability study needs performance goals appropriate to that tier. For example, remedy selection tests are used to answer the questions, "Will solvent extraction reduce contaminant concentrations to meet cleanup goals?" and "Can the concentrated contaminant be treated or reclaimed in a cost-effective manner?" A contaminant reduction of approximately 90 to 99 percent indicates that the technology may be able to meet cleanup goals and should be considered for the ROD.

The ideal technology performance goals are the cleanup criteria for the site. For several reasons, such as ongoing waste analysis and ARARs determination, cleanup criteria are sometimes not finalized until the ROD is signed, long after treatability studies must be initiated. Nevertheless, treatability study goals need to be established before the study is performed so that the success of the treatability study can be assessed. In many instances, this may entail an educated guess as to what the final cleanup levels may be. In the absence of set cleanup levels, the RPM can estimate performance goals for the treatability studies based on the first four criteria listed at the beginning of subsection 3.2. Previous treatability study results may provide the basis for an estimate of the treatability study goals when site cleanup goals have not been set.

#### 4.1.1 Remedy Screening Goals

Generally, the prescreening will be sufficient to determine the applicability of solvent extraction as the remedy or as a segment of the treatment train for a particular site. If the contaminants of concern include organics, then solvent

extraction can be considered a potential means of concentrating the organics. If the contaminants of concern do not include organics, then the solvent extraction processes referred to in this guide would not generally be applicable.

Remedy screening tests might be appropriate in an unusual sample such as a matrix which has not previously been extracted (e.g., peat or organic debris). Remedy screening may also be needed when a wide variety of contaminants are present in the matrix.

An example of the goal for those remedy screening tests would be to show that the chosen fluid is compatible with and will extract contaminants up to the clean up level if known or a sufficient percentage (e.g., 50 to 70 percent) to warrant further treatability studies to optimize the process. The remedy screening treatability study goals must be determined on a site-specific basis.

Achieving the goals at this tier should merely indicate that solvent extraction has at least a limited chance of success and that further studies will be useful. Occasionally, such information is available based on the type of contaminants and media present at the site and the availability of a compatible solvent at low cost. When such information is available, experts in solvent extraction technology can often assess the potential applicability of solvent extraction without performing remedy screening.

Example 1 describes a hypothetical site and a series of laboratory extraction tests that were used to evaluate the potential of solvent extraction for site remediation. The example illustrates how to decide whether the remedy selection treatability studies using solvent extraction should be performed.

#### 4.1.2 Remedy Selection Treatability Study Goals

The main objectives of this tier of testing are to:

- Measure the final contaminant concentration in and the percentage of contaminant removal from the soil, sludge, or water through solubilization in the chosen solvent(s).
- Produce the design information required for the next level of testing, should the remedy selection evaluation indicate remedy design studies are warranted.
- Provide cost estimates for full-scale remediation.

The actual goal for removal efficiency must be based on site- and process-specific characteristics. The specified removal efficiency must meet site cleanup goals, if available. A typical removal efficiency of 90 to 99 percent may be established for the remedy selection tier depending on the specifics of the site and the established cleanup goals.

Example 2 illustrates the goal of a remedy selection treatability study at the Superfund site introduced in Example 1. In this example, the remedy selection treatability studies show that site cleanup goals can be met. Solvent extraction is chosen as the selected remedy in the ROD.

## 4.2 EXPERIMENTAL DESIGN

### 4.2.1 Remedy Screening Tier

Screening tests can be rapidly performed in onsite or offsite laboratories using standard laboratory glassware or specially designed laboratory-scale extractors to evaluate the potential performance of solvent extraction as an alternative technology. Careful planning of experimental design and procedures is required to produce adequate treatability study data. The experimental design must identify the critical parameters and determine the number of replicate tests necessary.

When assessing the need for laboratory extraction tests, the investigator should use available knowledge of the site and any preliminary analytical data on the type and concentration of contaminants present. In general, the physical properties of solid and liquid media are important to the success of solvent extraction. Viscosity is critical to processes which require a pumpable feed material. Specific gravity affects phase separation. Particle size and pore space can influence the solvent's ability to extract the contaminants from the soil.

Contaminant characteristics to examine during remedy screening include solubility in various solvents. Vapor pressure and Henry's law constants are useful for evaluating solvent recovery methods. Properties of organic contaminants are generally easier to evaluate than those of inorganic contaminants. Inorganics, such as heavy metals, can exist in many compounds (e.g., oxides, hydroxides, nitrates, phosphates, chlorides, sulfates, and other more complex mineralized forms) which can greatly alter their solubilities. Inorganic leaching agents may be applicable for metal separation and removal.<sup>(4)</sup> Metal analyses typically provide only total metal concentrations. More detailed analyses to determine specific anions and cations present may be warranted.

At this level of testing the experimental design does not have to be vendor-specific. A recommended remedy screening test for contaminated soils is as follows:

- Both a hotspot sample and a "representative" or near averagesample of approximately 5 kg (see subsection 4.4.1) are placed in individual containers with a solvent at a soil-to-solvent ratio of approximately 1:5.
- Each container is thoroughly agitated for 2 hours using a rotary shaker or other device.
- After settling, each soil/solvent mixture is centrifuged.
- The solvent is decanted and sampled from each container.
- The soil from each container is centrifuged again, vacuum filtered, and sampled.
- Analyses of each decant and residual are performed.

A second option is using a soxhlet extraction for remedy screening. If a soxhlet is used, less than 1 kg of sample is required. In any case, remedy screening tests are generally run at ambient conditions with selected solvents from a

## Example 1. Remedy Screening

### BACKGROUND

A site which had been used for disposal of oily wastes for over 40 years was in the RI/FS stage of remediation. The wastes were stored in piles, pits, and lagoons. Data from the RI showed that throughout the site there was contamination with significant levels of semivolatile organic compounds (SVOCs). The concentration and composition of volatiles and metals varied considerably, as did the physical consistency of the solids and sludges. Samples also showed scattered, low concentrations of PCB's. Total oil and grease varied from 3 to 25 percent. Solids were apparently catalyst fines, clay, and carbon from refinery wastes; metals and carbon from used oil; and soil.

Because of the high levels of SVOCs, the material appeared to be a good candidate for solvent extraction. However, since no data was available on the extraction of waste mixtures of a similar composition and consistency a screening study was recommended.

### TESTING

The remedy screening study was recommended by the contractor to demonstrate the potential effectiveness for extracting the mix of oils and PCBs from sludge, and the semivolatiles from soil and fine solids. The project manager agreed to the testing. Two samples were selected for testing. These samples represented the extremes in the moisture content and physical characteristics of the soil. The first sample was a sludge from an area where PCBs had been detected. This sample contained primarily coarse soil particles. The other sample was from a "dry" pile containing a large percentage of fine soils. Approximately 200 grams of each sample was extracted with liquefied propane in a bench-scale extractor equipped with a magnetically driven mixer. In each case the sample was extracted in "4-stages" with a 2:1 solvent:feed ratio (by weight). This was done by placing the sample in the extractor, filling the extractor with propane, mixing for 10 minutes, settling, decanting off the propane solution, refilling with clean propane, and repeating the above cycle for four extractions.

The sludge, which contained about 40 percent water, was air dried and analyzed. The initial oil and grease content was approximately 25 percent. Oil and grease were reduced by about 96 percent and PCB's were non detectable in the solid residue. The extracted oil was also analyzed for heating value and PCB content. The heating value was 14,000 BTU/lb and the PCB's were 30 parts per million (ppm).

The "dry" pile sample was also extracted with propane in a bench scale batch extractor. The solids appeared to be a clay filter cake containing about 18 percent oil. After extraction the residual oil on the solids was approximately 0.4 percent, or a 98 percent reduction.

Solvent extraction was recommended for the follow up work. Since the screening study results were favorable, the need for a remedy selection treatability study was debated. However, since the site characteristics varied so greatly, it was decided to undertake a remedy selection study to test the solvent extraction process with a variety of contaminant/matrix mixes. The extracted oil sample was given to a rerrefiner to evaluate the potential to reclaim the recovered oil.

generic list. The test should be run using a hydrophilic solvent and then the residual solids from the first extraction should be subjected to a second extraction with a hydrophobic solvent. Hydrophilic solvents include acetone, methanol, and dioxane. Hydrophobic solvents include hexane and kerosene. CST solvents, such as triethylamine, can be either hydrophilic or hydrophobic depending on the temperature; however, such solvents are not generally used for remedy screening. The concentration of the contaminants of concern in the received soil, each solvent, and the treated soil is

determined.

When performing the remedy screening test, observe whether an emulsion forms, either at the top or the bottom of the container. Determine the settling time, settling rate, and depth of the solids. The rate and the relative volume of the settling material will provide some indication of the potential for solids separation. Removal efficiency can be estimated by analyzing the separated solids for selected indicator contaminants of concern. The removal efficiency goals for remedy screening should not

## Example 2. Remedy Selection

### BACKGROUND

The site discussed in Example 1 was recommended for additional remedy selection studies due to the variety of site matrix characteristics. The solvent extraction process had demonstrated its ability to remove a substantial percentage (>96 percent) of contaminant from two different media. However, the ability of the process to handle all of the solids and sludges in combination with oils and other contaminants in one processing system required verification. This opportunity was also used to demonstrate the technology onsite at the pilot scale, and to collect remedy design data.

### TESTING

A total of 12 samples representing the different matrices and contaminants was taken. Each of the 12 samples was individually extracted. Then a number of composites were made between sludges and dry solids in an attempt to simulate a homogeneous feed which could be maintained during the remediation by blending feed sources. These composites were extracted and used to test various processing parameters and to test the process at a larger scale.

Each of the twelve samples was extracted at the vendor's laboratory in the same type of bench-scale extraction equipment used in the screening tests. This required approximately 200 grams of each sample. The same basic test was run on each sample, that is "4-stages" with a 2:1 solvent:feed ratio (by weight). The total oil and grease was measured on each sample before and after extraction.

Next, three composites, each amounting to several gallons of sample, were made. Each composite had approximately the same ratio of liquids:solids, 70:30. The composites each included four different samples. Water was added to one sample to "liquefy" the sludge. Then in the same bench-scale extraction system, a series of tests was run on the composite to determine: likely operating conditions, the ability of the process to routinely meet cleanup goals, and approximate cleanup processing costs.

The extraction process variables which were tested included: solvent - pure propane and two different propane-butane blends; temperature - two temperatures, 65°F and 100°F, each at sufficient pressure to maintain the solvent completely liquefied; and solvent to feed ratio - 1:1, 2:1, and 4:1 on a weight basis. Since the samples were viscous, high intensity mixing was used in all tests. The total oil and grease, water, and solids was measured on each sample before and after extraction in the vendor's laboratory. Solid, water, and oil material balances were calculated for each test, with a quality assurance goal of 90 percent closure on each component.

be as stringent as those for remedy selection. Goals will, in general, be site- and contaminant-specific. If the cleanup level (if known) is attained or a significant removal efficiency (e.g. > 50 to 70 percent) is achieved for a given site during remedy screening, then solvent extraction can be viewed favorably and more detailed laboratory and bench tests must be conducted.

To reduce analytical costs during the remedy screening tier, a condensed list of known contaminants should be selected as indicators of performance. The selection of indicator analyses to track during remedy screening testing should be based on the following guidelines:

- 1) Select one or two contaminants that are most toxic or most prevalent.
- 2) Select indicator compounds to represent other chemical groups if they are present in the soil (i.e., volatile and semi-volatile organics, chlorinated and nonchlorinated species, etc.).
- 3) If polychlorinated biphenyls (PCBs) and dioxins are known to be present, select PCBs as indicators in the tests and analyze for them in the solids fraction. (A TSCA R&D permit is required for treatability studies on materials which contain greater than 50 parts per million (ppm) of PCBs.)

### Example 2. (continued)

Finally, approximately 50 gallons of each of the three composites were prepared onsite. This material was then extracted in a small portable pilot plant system brought to the contaminated site. The pilot plant included most of the operations which are in the full-scale system. However, it can be operated and brought to steady-state conditions using much smaller sample volumes than the full-scale system. The pilot-plant extractor is a multi-stage continuous countercurrent mixer settler, and includes a solvent recovery system. The mixer volume is approximately 20 gallons; at a 2:1 solvent:feed ratio by weight (4:1 by volume) there are approximately 4 gallons of sludge in the mixer at any one time. Thus the 50-gallon sample is sufficient for reaching and maintaining steady state for the bulk of the extraction time.

Each of the 50 gallon composites was extracted in this pilot plant in approximately 2 hours of continuous operation. Samples were taken from the feed and at the discharge of each extraction stage every 15 minutes during the test. In addition, after the first 8 gallons of extraction residue (two extractor volumes) were removed, the remaining residue was collected and composited for sampling and analysis. The extracted oil was also collected and composited for performance testing and analysis. The samples taken every 15 minutes were analyzed for oil and grease content to determine the length of time required to reach steady state, and to ensure that steady state was maintained. These sample analyses were also used to determine extraction stage efficiencies and in the calculation of the oil material balances. Three samples were taken from the composite extraction residue and sent to an independent test laboratory for analysis of total petroleum hydrocarbons, volatile, acid and base/neutral extractable and semivolatile organic compounds, and PCBs. After sampling and analysis, the three oil extract samples collected from each of the large-scale composite extraction tests were composited and rerefined to determine the potential for oil recycle.

## RESULTS

The results of the study indicated that with proper pretreatment, primarily blending, all of the waste matrices present could be extracted well below the target cleanup goals which had been tentatively set. Pretreatment was required to make all of the feed material approximately the same ratio of solids to liquids. This was accomplished in testing by blending the dry wastes with the sludges; alternatively it could be accomplished by slurring the dry wastes and partially drying the sludges.

The 12 samples tested showed oil extraction varying from 92 to 99 percent. The process tests showed that extraction in excess of 99 percent could be routinely achieved with a heavier solvent mixture and higher solvent-to-feed ratios than that used in the screening tests. The multiple samples and analyses run during the continuous extraction as well as the material balances met the quality assurance goals.

The data collected was used to determine the ability to consistently meet projected cleanup goals, to complete a preliminary process design for the cleanup, and to estimate the cleanup cost.

It is usually not cost-effective to analyze for all contaminants at this level of testing. Check for other contaminants later in the solids or water fraction from remedy selection tests. Once guidelines 1 through 3 have been applied, solvent(s) should be selected which are likely to extract the contaminants to be measured.

### 4.2.2 Remedy Selection Tier

This series of tests may use the same equipment as the remedy screening tier or may require additional equipment. The tests are run under more controlled conditions than the remedy screening tests. The removal efficiency is measured under variable extraction conditions

which can include the addition of several solvents or an entrainer; sequential extraction; heated solvents; pH adjustment; and use of supercritical or near-critical conditions. More precision is used in weighing, mixing, and phase separation. There is an associated increase in QA/QC costs. Wet soils and sediments may require dewatering before treatment. Chemical analyses are frequently performed on the solvent fraction as well as on the cleaned solids fraction. The impact of process variables on extraction efficiency is quantified. This series of tests is considerably more costly than remedy screening tests, so only samples showing promise in the remedy screening phase should be carried forward into the remedy selection tier. The objective of the remedy selection solvent extraction design is to meet the goals discussed in subsection 4.1.2.

Bench-scale testing is usually sufficient for this tier, but there are instances where additional pilot-scale testing is warranted. If foaming problems occurred during remedy screening or bench-scale testing, pilot-scale testing should be used to solve any problems before full-scale remediation. Pilot-scale testing may be necessary in order to obtain community acceptance. A pilot-scale or short-term run with full-scale equipment may be used for large sites in order to better define cost estimates for complete remediation.

A series of tests should be designed to provide information on the technical capability of solvent extraction to meet cleanup goals, as well as the cost of meeting the goals. The initial tests would typically consist of a few quick screening extractions similar to the one discussed in subsection 4.2.1 to determine the type of solvent system to be used, and to detect any unusual behavior or difficulties in the process. This would be followed by tests in which extraction variables such as solvent-to-feed ratio, extraction mixing intensity and time, number of stages, pH, temperature, and pressure would be examined. In order to optimize the field operating conditions, several test samples may be required for each variable. To hold down analytical costs, inexpensive screening analysis, such as only measuring initial and final TOC or TPH, could be used to indicate a relative percent removal. Only the final extraction test samples, running close to anticipated field processing conditions would be given full analyses. The full analyses are needed to verify the results of inexpensive screening analyses. In addition, the need or utility of pretreatment and posttreatment would be evaluated, and if appropriate, tested. The process data and analysis of samples should be of sufficient quality to allow estimates to be made of the cost of extraction as a function of cleanup level. The cost of pre- and post-treatment should also be evaluated along with the value or liabilities associated with the products of extraction.

Several factors must be considered in the design of solvent extraction treatability studies. A remedy selection test design should be geared to the type of system expected to be used in the field (i.e., standard solvents, critical fluids/liquefied gases, or CST solvents). Bench-scale testing does not have to be vendor-specific, but pilot-scale testing does. Solvent-to-feed ratios should be planned using the results from the laboratory screening tests, if they were performed. In general, solvent-to-feed ratios of 2:1 to 5:1 will be sufficient to perform remedy selection tests.<sup>(13)(17)(18)(19)(23)</sup> The solvent and solids should be mixed

for a minimum of 10 minutes and a maximum of 30 minutes. The solvent-to-feed ratio and mix times presented here are rules of thumb to be used if no other information is available.

Normally, only the solids fraction which has been cleaned and separated needs to be analyzed for contaminants. Contaminant concentration in the solvent may be determined periodically (e.g., 10 percent of the samples) to make an approximate material balance determination. Complete separation of the solids fraction from the solvent is necessary for accurate material balance calculations. Concentration measurements should be taken after each cycle or batch, or at timed intervals for continuous processes, so as to eventually be able to calculate the cost of removal versus the contaminant removal efficiency.

Initially, the solids fraction should be analyzed only for indicator contaminants. If the removal of the indicator contaminants confirm that the technology has the potential to meet cleanup standards at the site, additional analyses should be performed. Both the solvent fraction and the solids fraction must be analyzed for all contaminants if a complete material balance is desired. If any water is removed during the process, it should also be analyzed. A quantitative balance for volatile components may not be practical at this tier because of the cost of determining losses to the air.

The decision on whether to perform remedy selection testing on hot spots or composite samples is difficult and must be made on a site-by-site basis. Hot spot areas should be factored into the test plan if they represent a significant portion of the waste site. However, it is more practical to test the specific waste matrix that will be fed to the full-scale system over the bulk of its operating life. If the character of soils or sediments changes radically (e.g., from clay to sand) over the depth of contamination, then tests should be designed to separately study system performance on each soil type. Sample size for this tier of testing depends on the size of the test equipment and the number of test samples. Additional guidance on soil sampling techniques and theory can be found in Soil Sampling Quality Assurance User's Guide<sup>(34)</sup> and Methods for Evaluating the Attainment of Cleanup Standards.<sup>(31)</sup>

### 4.3 EQUIPMENT AND MATERIALS

The Work Plan should specify the equipment and materials needed for the treatability test. For example, the size and type of glassware or containers to be used during the test should be specified. Standard laboratory methods normally dictate the types of sampling containers which can be used with various contaminant groups. The RPM should consult such methods for the appropriate containers to be used for the treatability studies.<sup>(37)</sup> Normally, glass containers should be used. Stainless steel can also be used with most contaminants. Care should be taken when using various plastic containers and fittings. Such materials will absorb many contaminants and can also leach plasticizer chemicals, such as phthalate, into the contaminant matrix. Appropriate methods for preserving samples and specified holding times for those samples should be used.

The following equipment is recommended for remedy screening solvent extraction tests:



## Basic Equipment

- Standard laboratory extraction equipment (e.g., soxhlet, separatory funnel, etc.) or specialized solvent extraction equipment (e.g., high-pressure systems for critical fluids)
- Top loading balance
- Timer
- Sample jars
- Filter or centrifuge
- Vacuum pump
- Magnetic stirrer

Typically, the equipment used in remedy selection tests is similar to that of remedy screening in the case of bench-scale testing and vendor-specific in the case of pilot-scale testing.

## 4.4 SAMPLING AND ANALYSIS

The Work Plan should describe the procedures to be used in field and treatability study sampling. The procedures to be used will be site-specific.

### 4.4.1 Field Sampling

A sampling plan should be developed which directs the collection of representative samples from the site for the treatability test. The sampling plan is site-specific. It describes the number, location, and volume of samples. Heterogeneous soils and sediments, variations in the contaminant concentration profile, and different contaminants in different locations in the site will complicate sampling efforts. If the objective of the remedy screening or remedy selection treatability tier study is to investigate the performance of solvent extraction at the highest contaminant concentration, the sample collection must be conducted at a "hot spot". This will require conducting a preliminary site sampling program to identify the locations of highest contaminant concentration. (This information is generated early in the RI process). If the types of contaminants vary throughout the site and contaminants are located in several media, extensive sampling may be required. If solvent extraction is being considered only for certain areas of the site, the sampling program may be simplified by concentrating on those areas.

If the objective of the remedy selection study is to investigate the use of the technology for a more homogenous waste (sludge, water, or homogeneous soil), an "average" sample for the entire site must be obtained. This will require a statistically-based program of mapping the site and selecting sampling locations that represent the variety of waste characteristics and contaminant concentrations present. The selection of sampling locations should be based on knowledge of the site. Information from previous soil and water samples, soil gas analysis using field instrumentation, obvious odors, or residues are examples of information which can be used to specify sample locations.

Chapter 9 of Test Methods for Evaluating Solid Waste<sup>(37)</sup> presents a detailed discussion of representative samples and statistical sampling methods. Additional sources of information on field sampling procedures can be found in Samplers and Sampling Procedures for Hazardous Waste Streams (November 1987), Annual Book of ASTM Standards,<sup>(3)</sup> NIOSH Manual of Analytical Methods (February, 1984),<sup>(22)</sup> and the EPA publications Soil Sampling Quality Assurance User's Guide<sup>(34)</sup> and Methods for Evaluating the Attainment of Cleanup Standards.<sup>(31)</sup> These documents should be consulted to plan effective sampling programs for either simple or complex sites.

The method of sample collection is site-specific. For example, drill rigs or hand augers can be used to collect samples, depending on the depth of the sample required and the soil characteristics. If the target contaminants are volatile, care should be taken if samples are composited to minimize the loss of volatile compounds. Retaining composite samples on ice is a good method of minimizing the loss of volatile compounds. Compositing is usually appropriate for soils containing non-volatile constituents. A discussion of the field sampling plan is given in subsection 5.1 of this document.

### 4.4.2 Waste Analysis

Subsection 2.2.3 detailed the physical data that are useful in characterizing the contaminants during the prescreening step. The key for successful solvent extraction treatability studies is to properly select the solvent based on the initial prescreening and additional contaminant characterizations. Important matrix characteristics include the pH of solids and liquids, soil particle size, soil pore size, soil moisture content, and the viscosity of liquids and sludges. The pH is important in determining the compatibility of solvents with different contaminants. The speciation of metal compounds may also be affected by soil pH. Particle size and pore size information can be used to select process designs and/or solvents for treatment of solids or sludges. The soil moisture content is an important consideration for materials handling and dewatering processes.

Standard analyses for contaminants at Superfund sites should identify the contaminants of concern. It is important to determine contaminant solubility in various solvents to give an indication of potential solvents for testing. Volatility will be an important consideration for materials handling. If high concentrations of volatiles are present, pretreatment (e.g., using soil vapor extraction) or collection and treatment of air emissions may be required. Metal speciation will be an important consideration in determining metal solubility. However, complete analyses for metal species using x-ray diffraction is quite expensive. Typically, less costly methods are used to determine the primary anions and cations present.

The spatial distribution and variations in the concentrations of contaminants will be important for the design of treatability studies. Complex mixtures of contaminants may be difficult to treat economically. A number of extraction stages and solvents may be required to successfully remove many contaminants. The cost of such a system may be prohibitive. Changes in contaminant

composition can cause dramatic changes in removal efficiencies.

#### **4.4.3 Process Control Sampling and Analysis**

For any solvent extraction system, the operating conditions within the extractor are monitored and controlled to ensure efficient extraction is taking place. Temperature and pressure in the extractor are measured. The devices used for these measurements include thermocouple and pressure-sensing units which provide direct read-out capabilities and/or may be tied to a recorder or computer controlled system. Feed flow, solvent flow, and solvent-to-feed ratio are also monitored to verify operating conditions. Feed data such as pH, temperature, and viscosity also may be useful. Operating conditions of auxiliary equipment such as coolers, heaters, dryers, compressors, and pumps are routinely monitored.

#### **4.4.4 Treatment Product Sampling and Analysis**

Solvent extraction is not a stand-alone process (see subsection 2.1.1). It generates residuals which must be further treated and disposed of properly. The primary residual is the concentrated contaminants which are typically removed as the still bottoms during solvent recovery. Because the nature of solvent extraction equipment and processes varies greatly between vendors, remedy design testing is frequently necessary to evaluate the type, quantity, and properties of residuals. The remedy design treatability testing tier will not be discussed in detail in this document.

The treated solids, still bottoms, and each of the other various waste streams (water, spent solvent, and oversize fraction) should be analyzed for the contaminants identified in the original soil analyses. In many cases, indicator contaminants, which are representative of a larger group of contaminants, can be analyzed in place of a full scan. Caution must be exercised in using indicator contaminants since solvent extraction efficiencies can vary from one contaminant to another. The process efficiency may be either understated or overstated when analyzing for indicator compounds.

If several solvent extraction studies are run to test the effects of operating parameters on removal efficiency, samples of each test should be taken of each test before and after solvent extraction. Typically, these tests are run in triplicate.

#### **4.4.5 Sampling and Analysis Plan (SAP) and Quality Assurance Project Plan (QAPjP)**

A SAP is required for all field activities conducted during the RI/FS. The SAP consists of the Field Sampling Plan and the QAPjP. This section of the Work Plan describes how the RI/FS SAP is modified to address field sampling, waste characterization, and sampling activities supporting

treatability studies. It describes the samples to be collected and specifies the level of QA/QC required. See Section 5 of this document for additional information on the SAP.

#### **4.5 DATA ANALYSIS AND INTERPRETATION**

The Work Plan should discuss the techniques to be used in analyzing and interpreting the data. The objective of data analysis and interpretation is to provide sufficient information to the RPM and EPA management to assess the feasibility of solvent extraction as an alternative technology. After remedy selection testing is complete, the decision must be made whether to proceed to the remedy design testing tier, to a full-scale solvent extraction remediation, or to rule out solvent extraction as an alternative. The data analysis and interpretation are a critical part of the remedy selection process.

Chemical analysis of the contaminants present and interpretation of data generated in the solvent extraction process apply to all three tiers of the solvent extraction treatability study. The analysis of process test variables is limited to remedy selection and remedy design studies.

The primary goal of the remedy selection solvent extraction treatability testing is to determine how well the treatment removes the contaminant(s). System performance is affected by process design variables, including solvent-to-solids ratio, number of extraction stages, type of mechanical agitation used, agitated contact time, extraction temperature and pressure, system pH, and solvents sequence if more than one solvent is used. Often, two or more of these variables may affect the results. The concentration of the target contaminant versus the number of extraction stages is commonly graphed to determine number of stages required. Graphs such as these are intended to show general trends. The trends may not be consistent on a pass-by-pass basis. The plot on Figure 4-1 is an example of when concentration appears to increase (passes 4 and 10). These inconsistencies are related to cross contamination within system hardware or limited analytical precision and accuracy.<sup>(23)</sup> Statistical analysis of the data can be performed using standard techniques to differentiate sources of change and interactions between these sources. For a detailed discussion of the analysis of variance (ANOVA) techniques, and other statistical methods refer to the document entitled Statistical Analysis of Groundwater Data at RCRA Facilities (Interim Final)<sup>(25)</sup> and Lentner and Bishop.<sup>(12)</sup>

#### **4.6 REPORTS**

The last step of the treatability study is reporting the results. The Work Plan discusses the organization and content of interim and final reports. Complete, accurate reporting is critical, because decisions about implementability will be partly based upon the outcome of the study. The RPM may not require formal reports at each treatability study tier. Interim reports should be prepared after each tier. Project briefings should be made to interested parties to determine the need and scope of the next tier of testing. To facilitate the reporting of results and comparisons between treatment alternatives, a

suggested table of contents is presented in the generic guide.<sup>(27)</sup> At the completion of the study, a formal report is always required.

Vendors may be reluctant to provide information about the nature of proprietary solvent(s). Nevertheless, this information is necessary for measuring contaminants in the solvent or assessing the risk associated with residuals containing solvent. RPMs should consider including a separate section, possibly as an attachment, for any confidential business information.

OERR requires that a copy of all treatability study reports be submitted to the Agency's Superfund Treatability Database repository. One copy of each treatability study report must be sent to:

Glenn Shaul  
MS 445  
U.S. Environmental Protection Agency  
Superfund Treatability Database  
ORD/RREL  
26 West Martin Luther King Dr.  
Cincinnati, Ohio 45268

## 4.7 SCHEDULE

The Work Plan includes a schedule for completing the treatability study. The schedule gives the anticipated starting date and ending date for each of the tasks described in the work plan and shows how the various tasks interface. The time span for each task accounts for the time required to obtain the Work Plan, subcontractor, and other approvals (e.g., disposal approval from a commercial TSDF); sample procurement time, if necessary; analytical

turnaround time; and review and comment period for reports and other project deliverables. Some slack time also should be built into the schedule to accommodate unexpected delays (e.g., bad weather, equipment downtime) without affecting the project completion date. The schedule is usually displayed in the form of a bar chart (Figure 4-2). If the study involves multiple tiers of testing, all tiers should be shown on one schedule. Careful planning before the start of the tests is essential. Depending on the review and approval process, planning can take up to several months.

Setup of the laboratory and procurement of necessary equipment and lab supplies for treatability studies may take a month. Depending on how rapidly laboratory results can be provided, analytical results can be available in less than 30 days. Shorter analytical turnaround time can be requested, but this will normally double the costs. Compounds such as pesticides and PCBs may require longer turnaround times due to the extractions and analyses involved. Depending on the objectives, the duration of treatability tests may be longer.

Interpretation of the results and final report writing may take up to 4 months, but this is highly dependent on the review process. Remedy screening typically takes 3 to 4 months to complete treatability testing and results reporting. It is not unusual for the remedy selection phase to take 11 or 12 months before treatability testing and results reporting can be completed.

## 4.8 MANAGEMENT AND STAFFING

The Work Plan discusses the management and staffing of

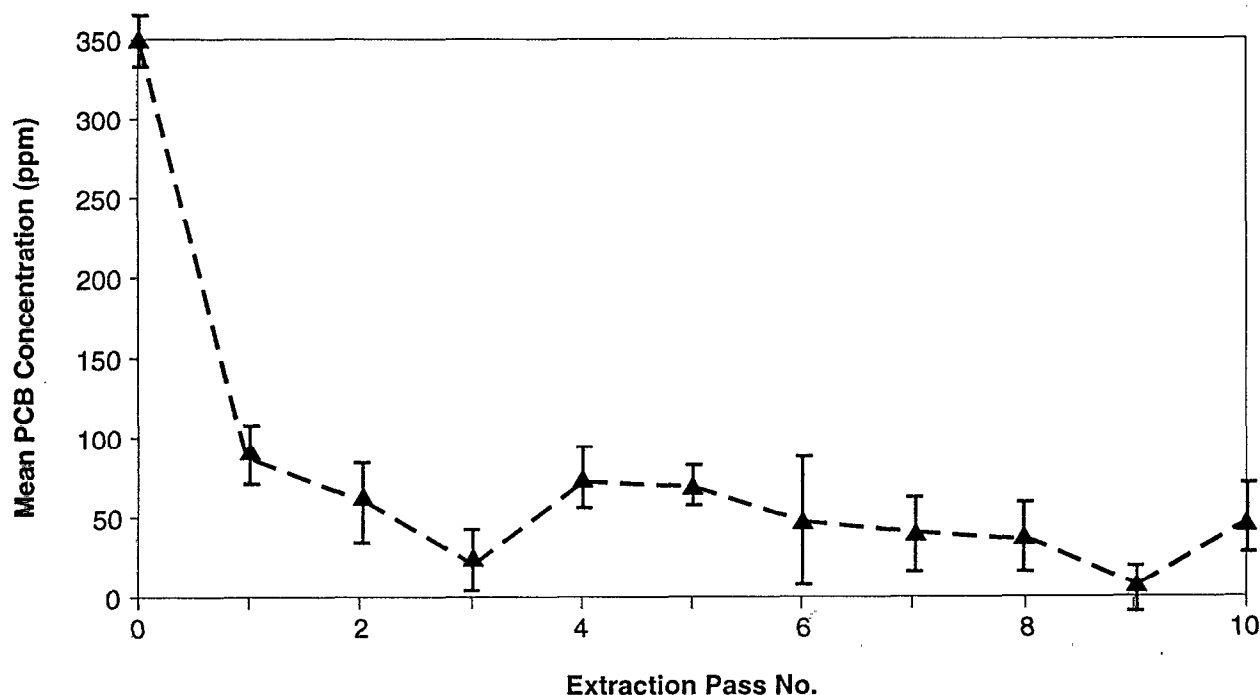


Figure 4-1 Example of pass-by-pass PCB concentration plot.

the remedy selection treatability study. The Work Plan specifically identifies the personnel responsible for executing the treatability study by name and qualifications. Generally, the following is an example of the types of expertise needed for the completion of the treatability study:

- Project Manager (Work Assignment Manager)
- QA Manager
- Chemist
- Chemical Engineer
- Lab Technician

Responsibility for various aspects of the project is typically shown in an organizational chart such as the one in Figure 4-3.

## 4.9 BUDGET

The Work Plan discusses the budget for completion of the remedy selection treatability study. Testing costs for remedy selection depend on a variety of factors. Table 4-2 provides a list of potential major cost estimate components for this tier. or most tests, the largest single expense is the analytical program. Sites where the soil and sediment types, contaminant types, and contaminant concentration vary widely will usually require more

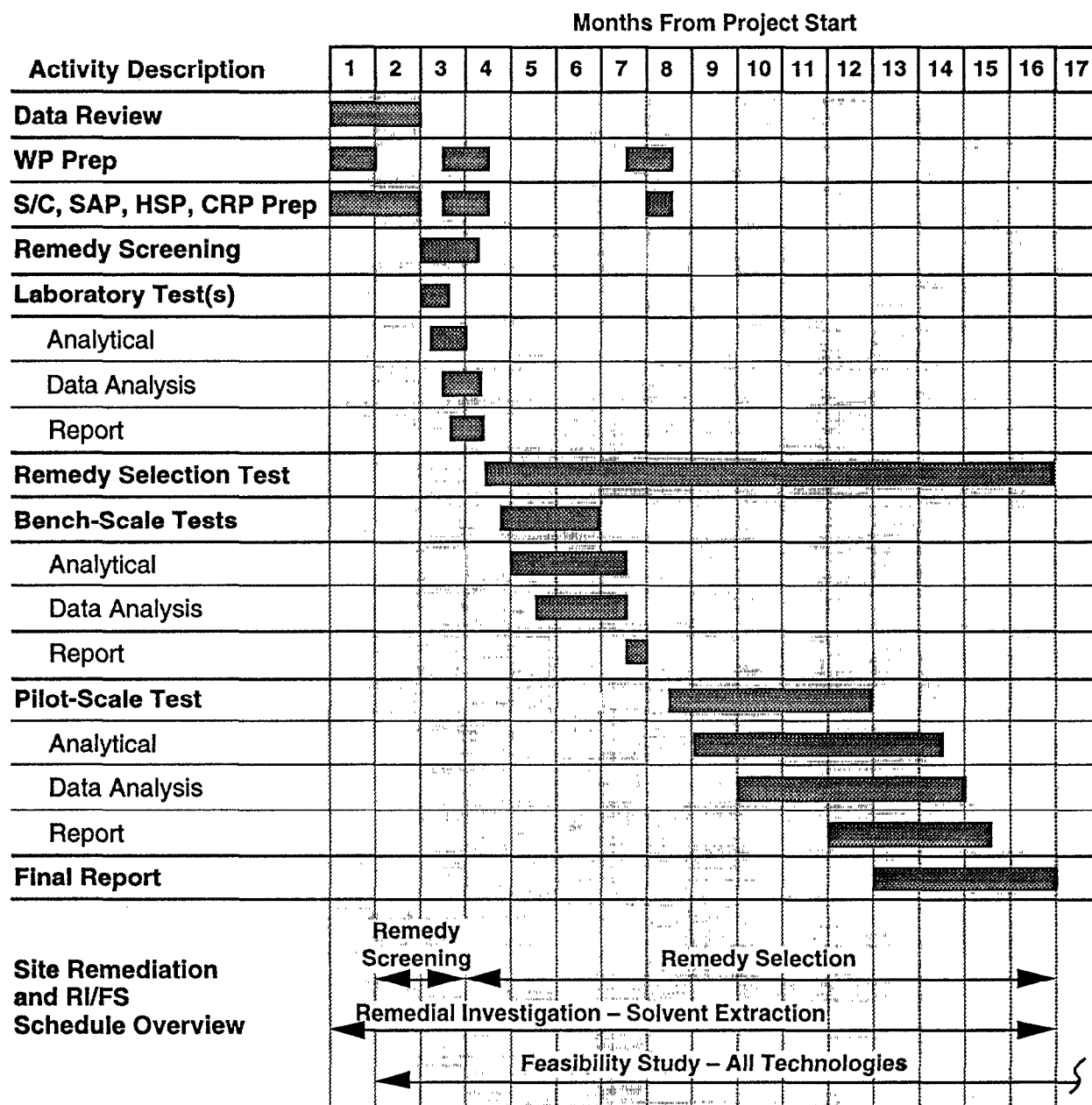


Figure 4-2. Example project schedule for a solvent extraction treatability study program.

samples than sites where the soil and contamination is more homogeneous. It is not unusual for the sampling, analysis, and QA activities to represent 50 percent of the total treatability study cost. In general, the costs for analyzing organics are more expensive than for metals. Actual costs will vary according to individual laboratories, required turnaround times, volume discounts, and any customized testing.

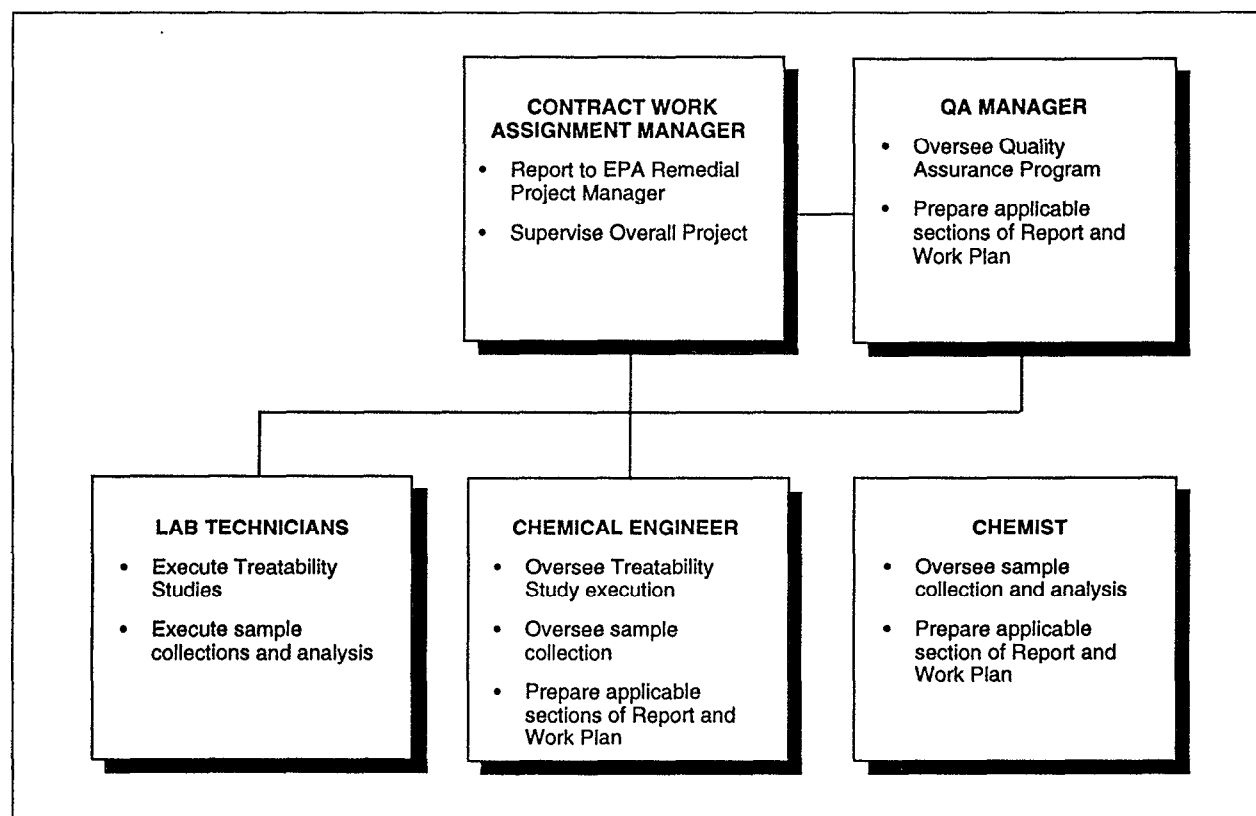
**Table 4-2. Major Cost Elements Associated with Remedy Selection Solvent Extraction Studies**

<b>Cost Element</b>	<b>Cost Range (thousands of \$)</b>
Initial Data Review	1 – 10
Work Plan Preparation	1 – 05
Field Sample Collection	1 – 10
Field Sample Chemical Analysis	4 – 25
Laboratory Setup/Materials/Testing	4 – 25
Treatability Test Chemical Analysis	4 – 20
Data Presentation/Report/Remediation Cost Estimate	5 – 25
<b>TOTAL COST RANGE</b>	<b>20 – 120</b>

Sampling costs will be influenced by the contaminant types and depth of contamination found in the soil, sludge, or sediment. The health and safety considerations during sampling activities are more extensive when certain contaminants, e.g., volatile organics, are present. Level B personal protective equipment (PPE) rather than Level D PPE can increase the cost component an order of magnitude. Sampling equipment for surface samples is much less complicated than equipment for deep samples. Depending on the number of samples and tests specified, residuals management (e.g., contaminated solvent and water) will require proper treatment and/or disposal. Treatment and disposal of the residuals as hazardous wastes increases costs significantly.

Other factors to consider include report preparation and the availability of vital equipment and laboratory supplies. Generally, an initial draft of the report undergoes internal review prior to the final draft. Depending on the process, final report preparation can be time-consuming as well as costly. Procurement of specialized testing equipment (e.g., bench-scale pressurized system) and laboratory supplies (e.g., reagents and glassware) will also increase the costs.

Typical costs for remedy selection tests are estimated to be from \$20,000 to \$120,000. The cost of remedy screening, with its associated lack of replication and detailed testing, is approximately 25 percent of these costs. These estimates are highly dependent on the factors discussed above. Not included in these costs are the cost of governmental procurement procedures, including soliciting for bids, awarding contracts, etc.



**Figure 4-3. Example organizational chart.**

## SECTION 5

# SAMPLING AND ANALYSIS PLAN

The Sampling and Analysis Plan (SAP) consists of two parts—the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPjP). The purpose of this section is to identify the contents of and aid in the preparation of these plans. The RI/FS requires a SAP for all field activities. The SAP ensures that samples obtained for characterization and testing are representative and that the quality of the analytical data generated is known and appropriate. The SAP addresses field sampling, waste characterization, and sampling and analysis of the treated wastes and residuals from the testing apparatus or treatment unit. The SAP is usually prepared after Work Plan approval.

### 5.1 FIELD SAMPLING PLAN

The FSP component of the SAP describes the sampling objectives; the type, location and number of samples to be collected; the sample numbering system; the equipment and procedures for collecting the samples; the sample chain-of-custody procedures; and the required packaging, labeling and shipping procedures.

Field samples are taken to provide baseline contaminant concentrations and contaminated material for treatability studies. The sampling objectives must be consistent with the treatability test objectives.

The primary objectives of remedy selection treatability studies are to evaluate the extent to which specific chemicals are removed from soils, sediments, sludges or water. The primary objectives for collecting samples to be used in remedy selection treatability testing include:

- Acquisition of samples representative of conditions typical of the entire site or defined areas within the site. Because a mass balance is required for this evaluation, statistically designed field sampling plans may be required. However, professional judgment regarding the sampling locations may be exercised to select sampling sites that are typical of the area (pit, lagoon, etc.) or to appear to have above average concentrations of contaminants in the area being considered for the treatability test. This may be difficult because reliable site characterization data may not be available early in the remedial investigation.
- Acquisition of sufficient sample volumes necessary for testing, analysis, and quality assurance and quality control. For remedy screening, about 5 kg will be

required. During remedy selection, the amount of sample will depend on the size of the test and the number of test samples.

From these two primary objectives, more specific objectives are developed. When developing the more detailed objectives, consider the following types of questions:

- Should samples be composited to provide better reproducibility for the treatability test? This question, including the type of compositing, is addressed in subsection 4.4.1.
- Is there adequate data to determine sampling locations indicative of the more contaminated areas of the site? Have soil gas surveys been conducted? Contaminants may be widespread or isolated in small areas (hot spots). Contaminants may be mixed with other contaminants in one location and appear alone in others. Concentration profiles may vary significantly with depth.
- Are the soils homogeneous or heterogeneous? Soil types can vary across a site and will vary with depth. Depending on professional judgement, contaminated samples for various soil types may have to be taken to conduct treatability tests.
- Are contaminants present in sediments, sludges, or water? Different sampling methods must be used for these media.
- Is sampling of a “worst-case” scenario warranted? Assessment of this question must be made on a site-by-site basis. Hot spots and contaminants in different media may be difficult to treat. These should be factored into the test plan if they represent a significant portion of the waste site.

After identifying the sampling objectives, an appropriate sampling strategy is described. Specific items that should be briefly discussed and included are listed in Table 5-1.

### 5.2 QUALITY ASSURANCE PROJECT PLAN

The QAPjP consists of eleven sections. Since many of these sections are generic and applicable to any QAPjP and are covered in available documents,<sup>(24)(32)</sup> this guide will discuss only those aspects of the QAPjP that are affected by the treatability testing of solvent extraction.

**Table 5-1. Suggested Organization of Sampling and Analysis Plan**

**Field Sampling Plan**

1. Site Background
2. Sampling Objectives
3. Sample Location and Frequency
  - Selection
  - Media Type
  - Sampling Strategy
  - Location Map
4. Sample Designation
  - Recording Procedures
5. Sample Equipment and Procedures
  - Equipment
  - Calibration
  - Sampling Procedures
6. Sample Handling and Analysis
  - Preservation and Holding Times
  - Chain-of-Custody
  - Transportation

**Quality Assurance Project Plan**

1. Project Description
  - Test Goals
  - Critical Variables
  - Test Matrix
  - Project Organization and Responsibilities
2. QA Objectives
  - Precision, Accuracy, Completeness
  - Representativeness and Comparability
  - Method Detection Limits
3. Sampling Procedures and Sample Custody
4. Analytical Procedures and Calibration
5. Data Reduction, Validation, and Reporting
6. Internal QC Checks
7. Performance and System Audits
8. Calculation of Data Quality Indicators
9. Corrective Action
10. QC Reports to Management
11. References

**5.2.1 Experimental Description**

Section 1 of the QAPjP must include an experimental project description that clearly defines the experimental design, the experimental sequence of events, each type of critical measurement to be made, each type of matrix (experimental setup) to be sampled, and each type of system to be monitored. This section may reference Section 4 of the Work Plan. All details of the experimental design not finalized in the Work Plan should be defined in this section.

Items in this section include, but are not limited to the following:

- Number of samples (areas or locations) to be studied
- Identification of treatment conditions (variables) to

be studied for each sample

- Target compounds for each sample
- Number of replicates per treatment condition
- Criteria for technology retention or rejection for each type of remedy selection test.

The Project Description clearly defines and distinguishes the critical measurements from other observations and system conditions (e.g., process controls, operating parameters, etc.) routinely monitored. Critical measurements are those measurements, data gathering, or data generating activities that directly impact the technical objectives of a project. At a minimum, the determination of the target compound (identified above) in the initial and treated solid samples will be critical measurements for remedy selection tests. Concentrations of target compounds in all fractions and the amount of solvent recovered will be critical measurements for remedy design tests.

**5.2.2 Quality Assurance Objectives**

Section 2 lists the QA objectives for each critical measurement and sample matrix defined in Section 1. These objectives are presented in terms of the six data quality indicators: precision, accuracy, completeness, representativeness, comparability, and, where applicable, method detection limit.

**5.2.3 Sampling Procedures**

The procedure used to obtain field samples for the treatability study are described in the FSP. They need not be repeated in this section, but should be incorporated by reference.

Section 3 of the QAPjP contains a description of a credible plan for subsampling the material delivered to the laboratory for the treatability study. The methods for aliquoting the material for determination of chemical and physical characteristics such as bulk density or specific gravity, moisture content, contaminant concentration, etc. must be described.

**5.2.4 Analytical Procedures and Calibration**

Section 4 describes or references appropriate analytical methods and standard operating procedures for the analytical method for each critical measurement made. In addition, the calibration procedures and frequency of calibration are discussed or referenced for each analytical system, instrument, device, or technique for each critical measurement.

The methods for analyzing the treatability study samples are the same as those for chemical characterization of field samples. Table 2-1 presents suitable analytical methods. Preference is given to methods in "Test Methods for Evaluating Solid Waste", SW-846, 3rd. Ed., November 1986.<sup>(37)</sup> Other standard methods may be used, as appropriate.<sup>(2)(3)(30)</sup> Methods other than gas chromatography/mass spectroscopy (GC/MS) techniques are

recommended to conserve costs, when possible, at the remedy screening level.

### **5.2.5 Data Reduction, Validation and Reporting**

Section 5 includes, for each critical measurement and each sample matrix, specific presentation of the requirements for data reduction, validation and reporting. Aspects of these requirements are covered in subsections 4.5, 4.6, and 6.1 of this guide.

### **5.2.6 Quality Control Reports**

Section 10 describes the QA/QC information that will be included in the final project report. As a minimum, reports include:

- Changes to the QA Project Plan
- Limitations or constraints on the applicability of the data
- The status of QA/QC programs, accomplishments, and corrective actions
- Results of technical systems and performance evaluation QC audits
- Assessments of data quality in terms of precision, accuracy, completeness, method detection limits, representativeness, and comparability.

The final report contains all the QA/QC information to support the credibility of the data and the validity of the conclusions. This information may be presented in an Appendix to the report. Additional information on data quality objectives<sup>(24)</sup> and preparation of QAPjPs<sup>(32)</sup> is available in EPA guidance documents.



## SECTION 6

# TREATABILITY DATA INTERPRETATION

Proper evaluation of the potential of solvent extraction for remediating a site must compare the test results (described in subsection 4.5) to the test goals (described in subsection 4.1) for each tier. The evaluation is interpreted in relation to seven of the nine RI/FS evaluation criteria, as appropriate. The remedy screening tier establishes the general applicability of the technology. The remedy selection tier demonstrates the applicability of the technology to a specific site. The remedy design tier provides information in support of the evaluation criteria. The test objectives are based on established cleanup goals or other performance-based specifications (such as removal efficiency). Solvent extraction testing must consider the technology as part of a treatment train.

Subsection 4.6 of this guide discusses the need for the preparation of interim and final reports and refers to a suggested format. In addition to the raw and summary data for the treatability study and associated QC, the treatability report should describe what the results mean and how to use them in the feasibility study in both screening and selection of alternatives. The report must evaluate the performance of the technology and give an estimate of the costs of final remediation with the technology.

### 6.1 TECHNOLOGY EVALUATION

Remedy screening treatability studies typically consist of simple laboratory tests. The contaminant concentration in the solids fraction, or water before extraction, is compared to the contaminant concentration in the same fraction after extraction. A removal of approximately 50 to 70 percent of the contaminants during the test indicates additional treatability studies are warranted. Contaminant concentrations can also be determined for wastewater and solvent fractions. These additional analyses add to the cost of the treatability test and may not be needed. Before and after concentrations can normally be based on duplicate samples at each time period. The mean values are compared to assess the success of the study. A number of statistical texts are available if more information is needed.<sup>(5)(11)(12)</sup>

Remedy screening tests can sometimes be skipped when information about the contaminant solubilities in the selected solvent is sufficient to decide whether remedy selection studies will be useful. This information should be solvent- and contaminant-specific and may or may not be applicable to other sites. Expert assistance is needed for evaluation of data for a site. Example 3 demonstrates a prescreening evaluation and the decision to bypass a

remedy screening test.

The remainder of this section discusses the interpretation of data from remedy selection treatability studies. Subsections 4.1 and 4.2 of this guide discuss the goals and design of remedy selection treatability studies, respectively. Typically, contaminant concentrations in the contaminated matrix before and after solvent extraction are measured in triplicate. A reduction in the mean concentration to cleanup levels, if known, or by approximately 90 to 99 percent indicates solvent extraction is potentially useful in site remediation. A higher QA level is required with this tier of testing. A number of other factors must be evaluated before deciding to proceed to remedy design studies.

In scaling the cost and performance estimates from remedy selection testing to full-scale solvent extraction systems, the parameters for consideration are:

- Performance capabilities of the solvent extraction process including design parameters
- Residual contaminants and contaminant concentrations in the solids fraction
- Contaminants and contaminant concentrations in the used solvent, in the fine soils, and in the concentrated contaminant product
- Risk analysis evaluation for worker and community protection
- Quantity of oversized screenable material
- Amount of contaminated water generated in dewatering and distillation processes.

The design parameters for the solvent extraction process include material throughput and optimum solvent usage in gallons per dry ton of solids or gallon of water. It is important to estimate the volume and physical and chemical characteristics of each fraction to design treatment systems and estimate disposal costs. The ability to cost-effectively recover used solvent is also important for cost and performance estimates. Removal efficiency, measured as a function of the number of extraction stages, can be used to determine the number of stages required to reach cleanup levels.

The final concentration of contaminants in the recovered (clean) solids fraction, in the solvents, in solvent distillation bottoms, and in water fractions are important to evaluating the feasibility of solvent extraction. The selection of technologies to treat the solvent or solvent still bottoms

and water fraction from soil/sludges depends upon the types and concentrations of contaminants present. The amount of volume reduction achieved in terms of contaminated media is also important to the selection of solvent extraction as a potential remediation technology.

Contamination in excavated soils and sediments can pose safety concerns for workers and community. Worker protection may be required during soil excavation. The need for such protection is a site-specific decision. Health and safety plans should be prepared and a risk analysis conducted for the site.

The quantity of large rocks, debris and other oversize screenable material that must be removed is an important measurement. While this is not a "laboratory" measurement, it is important to determine which treatment method is most suitable for preparing the bulk soil or sediment for entry into the solvent extraction process, i.e., screening to remove large rocks, stumps, debris, and washing or crushing of oversize materials, etc. The quantity and degree of contamination of water is important for design of ultimate treatment systems. The water could be the media to be treated or could be associated with a soil/sludge media.

## 6.2 ESTIMATION OF COSTS

Accurate cost estimates for full-scale remediation are crucial to the feasibility study process and the subsequent detailed analysis of alternatives. Comparisons of various technologies must be based on the most complete and accurate estimates available. Remedy screening treatability studies cannot provide this type of information. However, preliminary cost estimates for full-scale remediation may be made from remedy selection data. Such estimates may be good enough for comparisons to other technologies at the same tier of testing. On this basis, the estimates can form the basis of the ROD. Pilot-

scale tests yield more accurate estimates of full-scale performance and costs. This is especially true since solvent extraction will form only one component of a treatment train. If the results of remedy selection treatability testing indicate that solvent extraction can be effective, consideration may be given to pilot-scale testing. The cost for pretreatment of media and post-treatment of contaminated solids, still bottoms, and/or water from the solvent extraction process must also be evaluated.

### 6.2.1 Solvent Extraction Pilot-Scale Cost Estimates

Pilot-scale tests can be used to obtain a preliminary cost estimate for full-scale remediation. Bench-scale does not give information on all major cost estimate components in a full-scale solvent extraction operation. The major cost estimate components which can be determined based on pilot-scale results and site characterization data are as follows:

- Analytical
- Excavation
- Material handling and transport
- Pretreatment
- Treatment cost and throughput
- Treatment and/or disposal of residuals.

### 6.2.2 Actual Full-Scale Solvent Extraction Cost Estimates

Full-scale solvent extraction cost estimates will be solvent- and site-specific. As of Spring 1991 only six sources of portable soil/sludge extraction units were identified:

#### Example 3. Decision to Bypass Remedy Screening

A harbor sediment was being considered for solvent extraction. The sediment was contaminated with medium to high levels of PCBs. The sediment samples had a consistency similar to many sludges which had been extracted in previous studies. Although the concentration of PCBs was the highest that had been observed in any of the RIs involving solvent extraction, treatability studies had been performed on samples with the same order of magnitude of PCB contamination.

The technology vendor and resident solvent extraction expert were confident that the remedy screening study could be passed over, and the remedy selection study started immediately to identify the level of removal which could be expected in the remedy design and the remediation. The RPM agreed, and the remedy selection study was designed and implemented.

	Approximate feed capacity <sup>1</sup>
CF System's process	0.2 tons/hour <sup>2</sup>
RCC's B.E.S.T.™ process	3 tons/hour <sup>3</sup>
ART's LEEP <sup>sm</sup> process	1 ton/hour
Nukem Development's process	ND <sup>4</sup>
Sanivan Group's Extraksol™	1 ton/hour
Terra Kleen's Soil Restoration Unit	2 tons/hour
Dehydro-Tech's Carver-Greenfield Process	ND <sup>4</sup>

<sup>1</sup> May vary depending upon feed material and contaminant concentration

<sup>2</sup> Modular system may be used to increase capacity

<sup>3</sup> 110 lb/day pilot unit also available

<sup>4</sup> Process feed capacity not determined

Cost estimates for full-scale solvent extraction range from \$90 to \$800/ton.<sup>(2,3)(28)</sup> These estimates were provided by various vendors. It was not possible to determine from the estimates the extent of pre-or post-treatment associated with the costs, the operating parameters of their equipment, or the target cleanup levels required at the associated sites.

Cost estimates with one commercial-size system were determined using a base case (880,000 tons of sediment

containing 850 ppm of PCB) to a hot spot case (63,000 tons of sediments containing 10,000 ppm of PCB). The base case cost estimate with pre- and post-treatment was \$148/ton using two 250-ton/day capacity units in parallel. The hot spot case cost estimate was \$447/ton using a 100-ton/day capacity system consisting of two modules in series, with each module containing extraction and solvent recovery units in series. Another vendor reported cost estimates of \$90/ton using a 200-ton/day facility. The cost for using a smaller facility treating 30 tons/day increased to \$280/ton. These projected costs are based on the use of 25 yd<sup>3</sup>/day modules. For another site, the vendor used operational experience to estimate that the cost of operating a 30-ton/day module could range from \$150 to \$800/ton.

General factors affecting full-scale cleanup cost for solvent extraction are, the contaminants of concern, the required cleanup levels at the site, and the specific type of equipment selected for use. Specific factors affecting costs include the number of cycles for continuous processes, the number of extraction stages for batch processes, the size of the site, the initial concentration of contaminant(s), the type of soil, the amount of oversized materials, the type of foreign materials in the soil (metal nuts and bolts, building debris, etc.), the distance to the site, requirements for further treatment of residuals, insurance required, and bonding required. The disposal options for process waste streams and laboratory requirements for process sample analysis will also affect costs. Potential cost factors such as field change orders issued will be undetermined until remediation has been initiated.

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